

TOXICITY OF CERTAIN HERBICIDES IN SOILS¹A. S. CRAFTS²

PREVIOUS PUBLICATIONS from this station have described a method for testing the toxicity of chemical sterilants in soils (Crafts, 1935)³ and have presented results from such tests with arsenic, borax, and chlorate (Crafts, 1935; Crafts and Raynor, 1936). Further studies by this method have provided data on the characteristics of ammonium thiocyanate, sodium thiocyanate, ammonium sulfamate, dinitro-*o*-cresol, and certain other soil sterilants. At times when arsenic and chlorate cannot readily be obtained, the results of these studies may be especially useful; some of the chemicals tested may offer possibilities as substitutes. Furthermore, the present extensive use of substituted phenols in weed control presents problems concerning the possible deleterious effects of their accumulation in soils. With the introduction of new and more toxic organic herbicides, the possibility of using these as soil sterilants seems worth considering.

TESTING METHOD

Briefly, the testing method involves growing indicator plants (Kanota oats) in soils moistened with solutions of the chemical being studied. Surface soils are pulverized, screened, and weighed into no. 2 cans. Each culture is then moistened enough to bring it to field capacity, the solutions being made up by diluting a stock solution. After standing overnight the cultures are seeded. The plants are grown for 30 days, being periodically watered by bringing the cultures up to their original moist weight.

In harvesting, a visual estimate is made of the average height of the plants; then they are cut off at the soil surface and weighed. All yield data in this paper are average values for fresh weight of three or more cultures at harvest time.

After weighing, the tops are returned to the can, and all cultures are dried for 30 days. Then the tops are set aside, the soil is removed and pulverized, the tops are placed in the bottom of the can, and the pulverized soil is poured back in and remoistened with tap water. The cultures are seeded, watered, and harvested as before.

The percolation tests are made in special soil tubes. Each tube consists of a celluloid liner supported by hardware cloth bent into the form of a cylinder. The bottom is closed with a circle of filter paper supported by a square of $\frac{1}{8}$ -inch-mesh hardware cloth. In the percolation studies, series of these tubes

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³ See "Literature Cited" for complete data on citations, referred to in the text by name of author and date of publication.

are filled with air-dry soil and moistened from above by allowing the test solutions to drop on them slowly from small glass pipettes. After standing overnight each tube is laid on a table and opened, and the soil column cut into ten equal fractions. The moist fractions are carefully transferred to no. 2 cans and seeded. Watering and harvesting are the same as in the toxicity tests.

CHEMICALS TESTED

Ammonium Thiocyanate.—A previous publication (Robbins, Crafts, and Raynor, 1942) has summarized earlier work with ammonium thiocyanate as a herbicide and has briefly reported the following tests. Data on the soil tests include results of three crops on ten California soils—Arbuckle clay loam, Stockton adobe clay, Aiken clay loam, Yolo sandy loam, Egbert loam, Fresno sandy loam, Yolo adobe clay, Columbia fine sandy loam, Yolo clay loam, and Sacramento clay. Figures 1 to 4 illustrate these results. The graphs show yields only to the 1,220 p.p.m. concentration level. Included in the test series were cultures having concentrations to 2,040 p.p.m.

In these tests the cultures were seeded on the same day that they were moistened with the ammonium thiocyanate solutions. Evidently the solutions were very toxic. The results differed from those with arsenic (Crafts, 1939*b*) in that little change occurred during the first few hours. Considering the first-run curves of figures 1 to 4, one finds a general relation between crop yield and toxicity. If the curves are examined in the order of increasing yields (approximately in the order in which the soils are listed above) it will be found that toxicity decreases in that order. Though some exceptions occur, the same relation holds generally in the second and third runs. This is also the relation that holds for chlorate toxicity in soils (Crafts, 1939*a*).

As shown by the curves for the second runs, toxicity has changed tremendously during the 30-day cropping period and subsequent 45-day drying period of these cultures. Whereas practically no plants survived at a concentration above 80 p.p.m. in the first run, most cultures had better crops at this concentration in the second run than they did in the untreated checks. Plants made fair growth in Yolo clay loam initially having 490 p.p.m. (fig. 4), and in Yolo adobe clay they weighed 1 gram per culture in cultures initially containing 1,220 p.p.m. (fig. 3).

The inverse relation between fertility and toxicity of ammonium thiocyanate is again apparent. There are three possible exceptions—Yolo adobe clay, Egbert loam, and Sacramento clay. The first is a heavy soil whose colloid was isolated by water separation. This colloid has a marked fixing capacity. While the first run was being made, the toxicity of thiocyanate was altered more in this soil than in any other. Since Egbert loam and Sacramento clay samples were high in organic matter, toxicity may result from a summation of the effects of thiocyanate and excess nitrate. Both these soils are extremely high in organic matter, and their curves drop steeply after a very short initial rise.

Curves for the third runs show that toxicity of the chemical continued to decrease. Five of the soils show greater yields at levels initially containing 920 p.p.m. than in the untreated checks, and four of these had similar yields at levels initially containing 2,040 p.p.m. Soils that still have toxic amounts

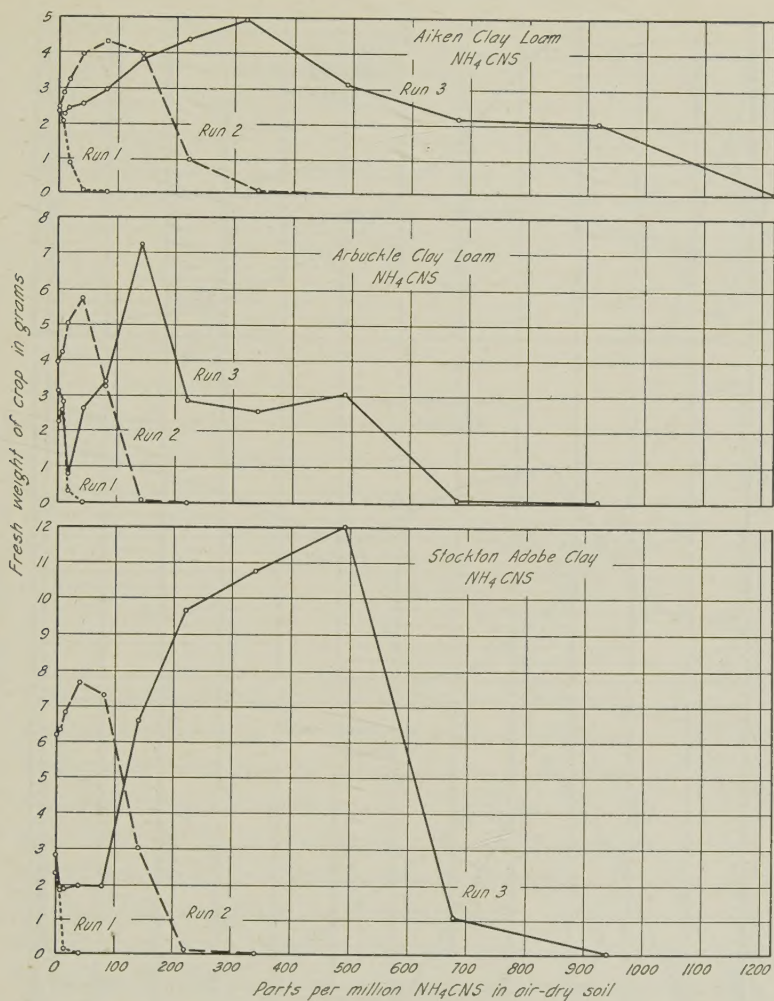


Fig. 1.—Toxicity of ammonium thiocyanate to indicator plants in Aiken clay loam, Arbuckle clay loam, and Stockton adobe clay.

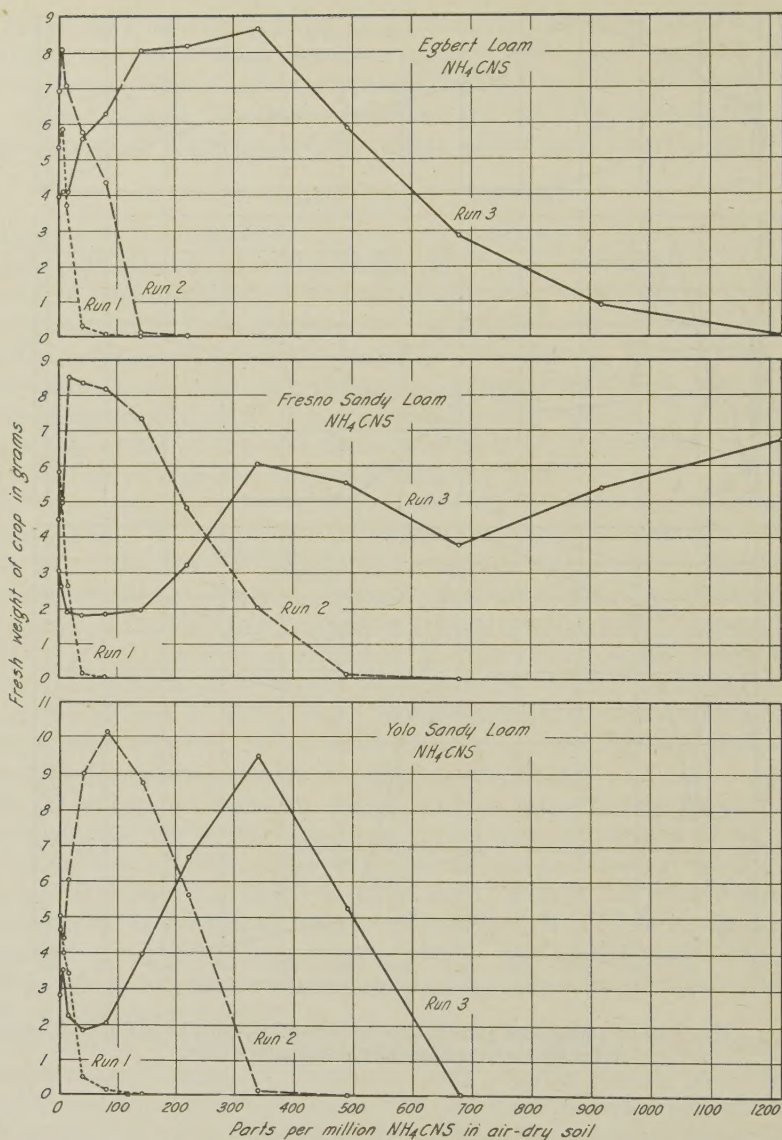


Fig. 2.—Toxicity of ammonium thiocyanate to indicator plants in Egbert loam, Fresno sandy loam, and Yolo sandy loam.

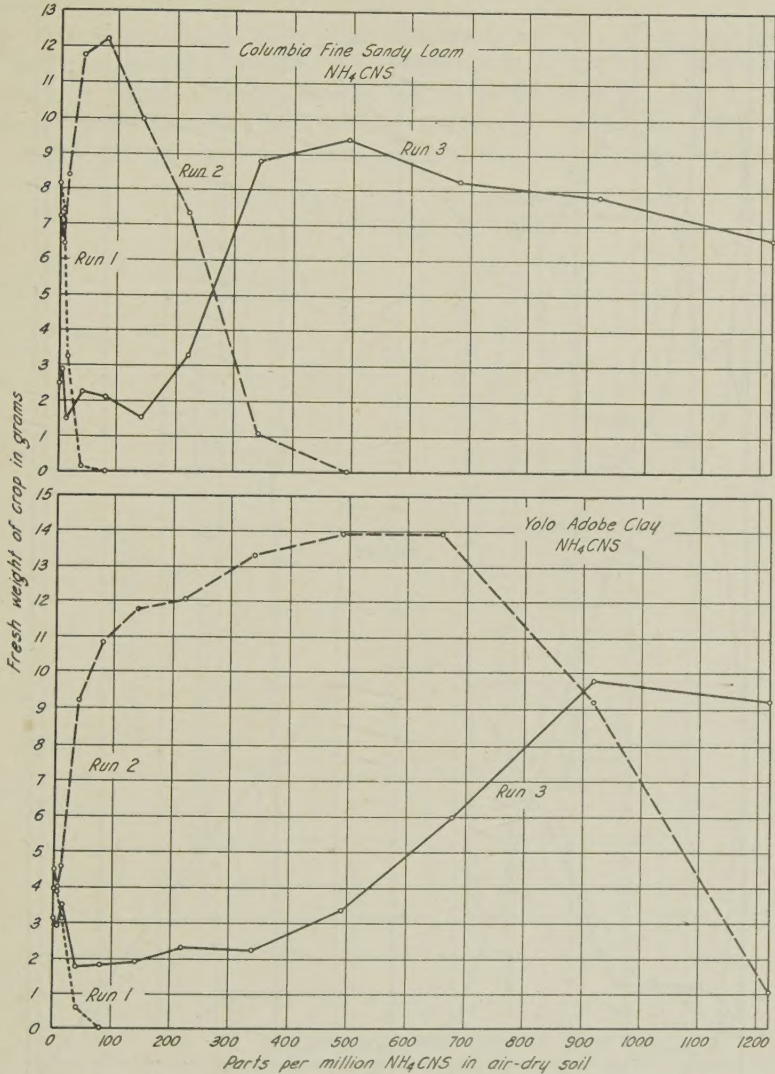


Fig. 3.—Toxicity of ammonium thiocyanate to indicator plants in Columbia fine sandy loam and Yolo adobe clay.

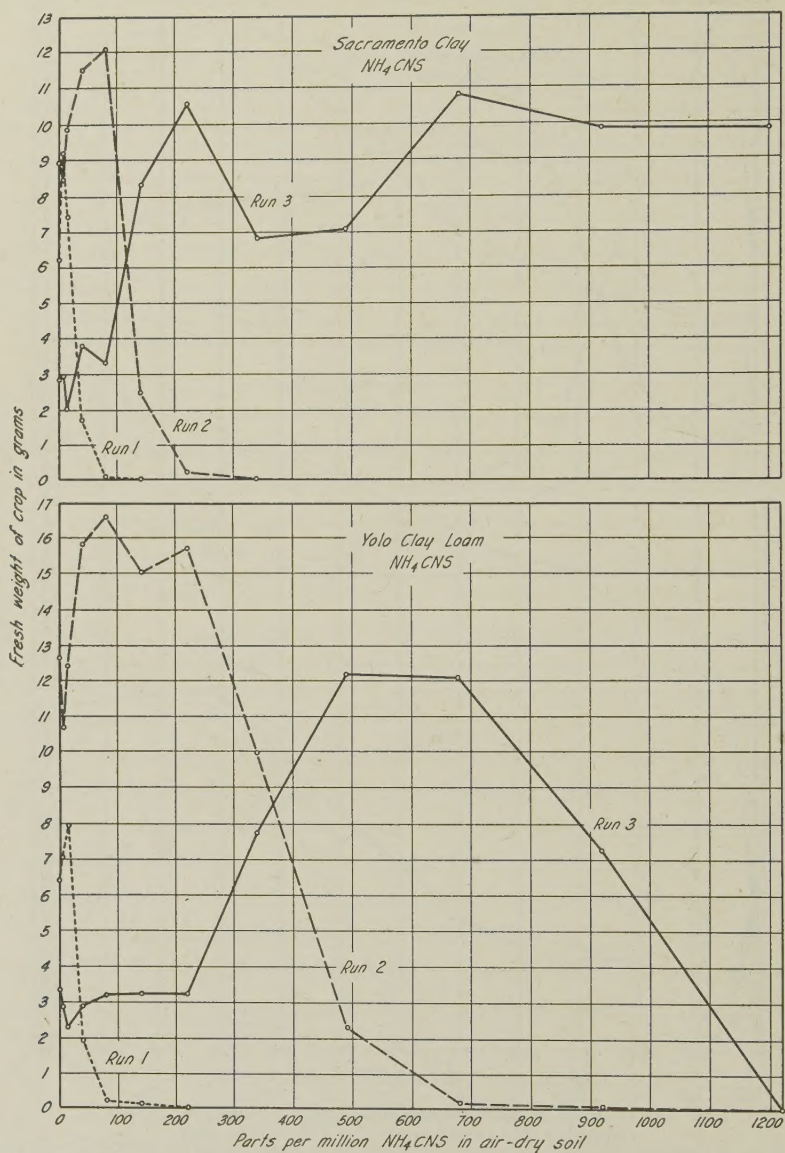


Fig. 4.—Toxicity of ammonium thiocyanate to indicator plants in Sacramento clay and Yolo clay loam.

of ammonium thiocyanate are Stockton adobe clay, inherently low in nitrogen; Yolo sandy loam, fairly light and low in colloid; Aiken clay loam, low in both nitrogen and available phosphate; and Egbert loam, in which excessively high nitrate might have contributed to the toxicity.

These curves have one interesting feature: the yields in the third run were low in the cultures that produced the greatest crops during the second run.

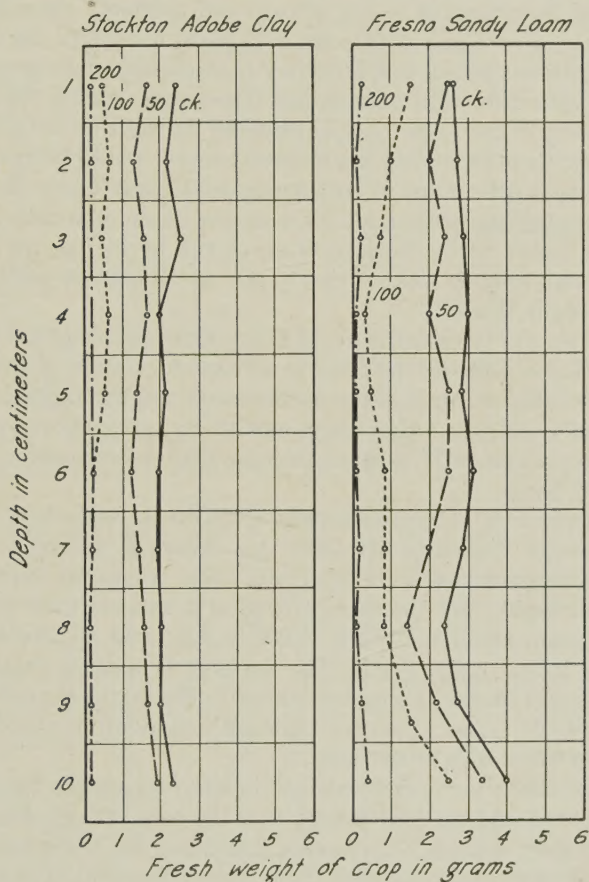


Fig. 5.—The relation of crop yield to the penetration of ammonium thiocyanate into columns of Stockton adobe clay and Fresno sandy loam. The chemical is not retained by these soils.

Evidently the nutrients tied up in the straw of the second crop were not released rapidly enough by decomposition of the straw to become available to the third crop. This effect is often noted in the field after large amounts of raw organic matter are plowed under.

The fourth runs showed no toxicity throughout the range tested—that is, from 0 application to cultures initially containing as much as 2,040 p.p.m. The fourth runs were planted December 13, 1937, almost a year after the initial planting.

Figure 5 gives the results of two percolation series with ammonium thiocyanate. As the graphs show, this chemical is not retained in an available form in the soil, but percolates down with the moisture as do chlorate (Rosenfels and Crafts, 1941) and nitrate.

According to the results just described, ammonium thiocyanate is extremely toxic when first applied to the soil; but thereafter it rapidly decomposes, losing its toxicity and contributing to the nitrate and probably the sulfate content of the soil. Since nitrate and sulfate are both plant nutrients, the decomposing ammonium thiocyanate acts as a fertilizer to stimulate plant growth.

The results also show that ammonium thiocyanate moves readily through the soil and hence would be subject to leaching by rains or irrigation. Apparently, therefore, its most logical use in weed control would be against annuals and shallow-rooted perennials in pastures or turf lands where the crop plants may rapidly occupy the areas made bare by the death of weeds. Utilizing the nutrients that result from decomposition of the herbicide, the crop should thrive and present greater competition to any seedlings that might grow from seed germinating in the soil.

Application should be timed to avoid heavy rains or irrigation immediately following, so that a sufficient interval is allowed for action of the toxicant.

The high toxicity of ammonium thiocyanate might suggest its use as a general contact herbicide. Other chemicals being tested, however, including the substituted phenols, offer so much promise that thiocyanates seem unlikely to compete with them.

Sodium Thiocyanate.—Figures 6 and 7 illustrate the effects of sodium thiocyanate upon soils. Tested in six soils, this chemical proved somewhat less toxic than the ammonium salt in the initial runs. It became much reduced in toxicity with cropping. By the third cropping it was nontoxic in four of the six soils at concentrations initially 1,220 p.p.m. and in the other two at concentrations initially 680 p.p.m. The toxicity of sodium thiocyanate, like that of the ammonium salt, seems unrelated to the textural grade of the soil. Relation to fertility is less apparent than with ammonium salt, but this may be only because fewer soils were tested.

Since sodium tends to replace calcium in the replaceable base complex of soils, with a corresponding deflocculation of the colloids and decrease of permeability to water, sodium thiocyanate could not be recommended for repeated use on western soils. Judging from tests in the field, it is not a particularly effective herbicide, either as a general contact spray or as a temporary soil sterilant. If it is used, the same recommendations apply as those outlined for the ammonium salt. Because of the lower toxicity, the dosage rate should be somewhat higher.

Ammonium Sulfamate.—Eleven soils were used in studying the toxicity relations of ammonium sulfamate—Aiken clay loam, Stockton adobe clay, Sierra fine sandy loam, Arbuckle clay loam, Yolo fine sandy loam, Willows adobe clay, Sacramento clay, Yolo adobe clay, Yolo clay loam, Hanford fine sandy loam, and Egbert loam. The first run was prepared and seeded on October 16 and 17, 1942. The tests were carried through four runs, terminating in mid-July of 1943.

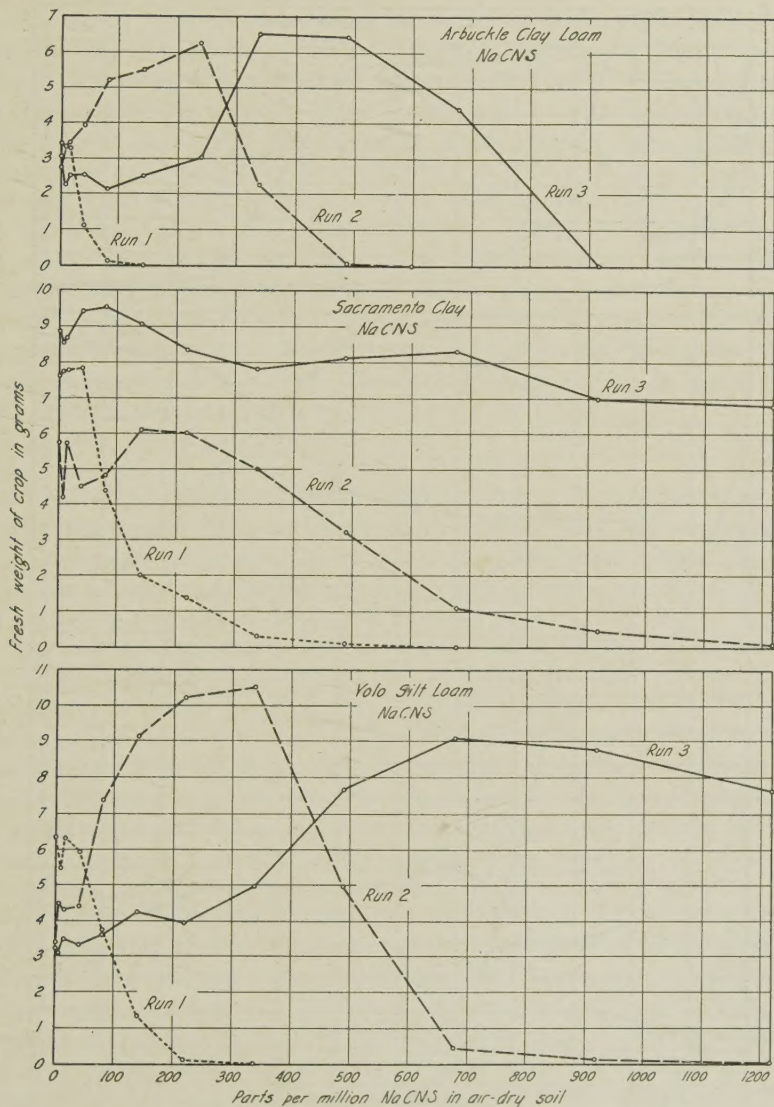


Fig. 6.—Toxicity of sodium thiocyanate to indicator plants in Arbuckle clay loam, Sacramento clay, and Yolo silt loam.

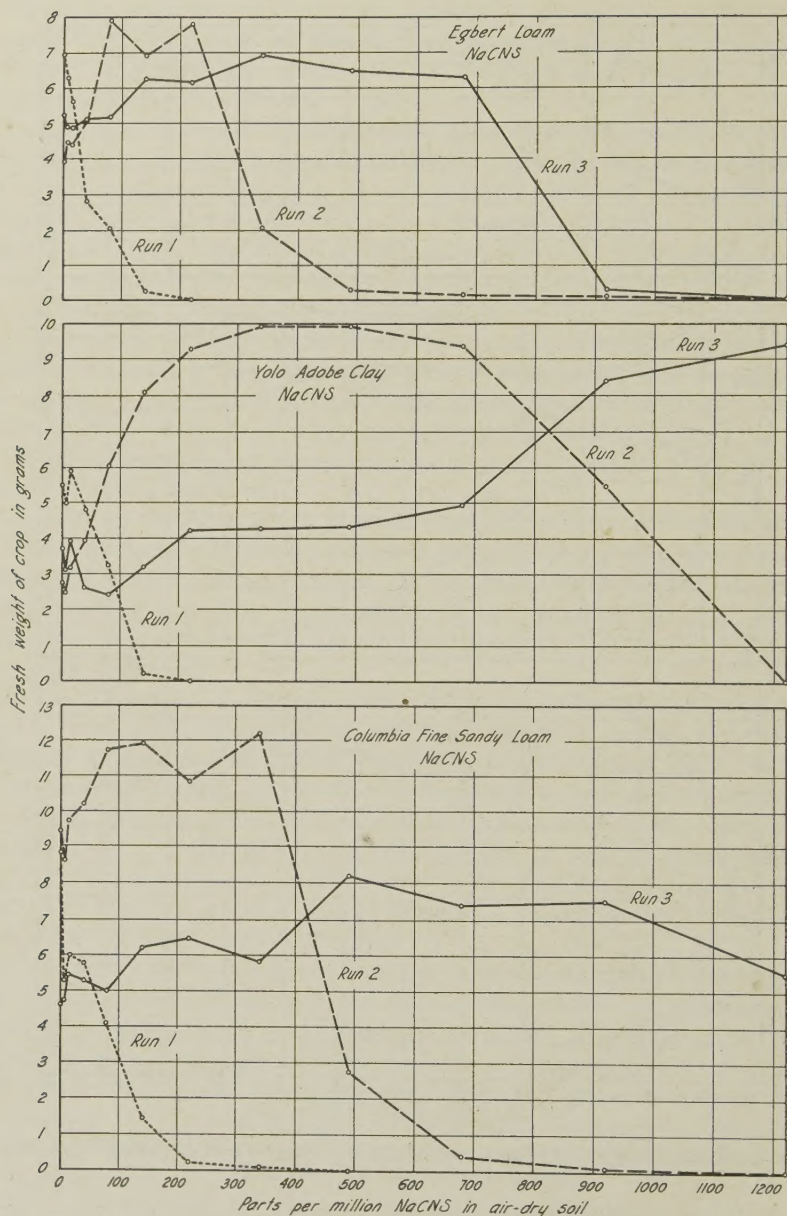


Fig. 7.—Toxicity of sodium thiocyanate to indicator plants in Egbert loam, Yolo adobe clay, and Columbia fine sandy loam.

Results of the toxicity tests (figs. 8, 9, and 10) show that sulfamate is much lower in initial toxicity than thiocyanate. When the curves are studied in relation to soil characteristics, toxicity again appears to be determined more by fertility than by textural grade. Egbert loam (fig. 9) is an exception. As with thiocyanate, its response to sulfamate may be complicated by its high initial content of nitrate. On the other hand, reports from eastern states indicate that sulfamate may be more toxic in acid soils; and Egbert loam, being very high in organic matter, has an acid reaction (Cosby, 1941).

By the time of the second runs, toxicity has markedly decreased. In most soils, concentrations that reduced growth to low values in the first run now have stimulated the yields to higher levels than those of the untreated checks. Only two soils, Aiken clay loam and Stockton adobe clay, are sterilized by concentrations initially below 500 p.p.m. Yolo adobe clay at a concentration initially 920 p.p.m. has a yield considerably higher than in the untreated check. Again toxicities are inversely related to fertility.

Curves for the fourth run show continued decrease in toxicity, but the change was much less marked than with thiocyanates.

An interesting feature of these toxicity curves is the indication of stimulation in the region of 15 to 40 p.p.m. during the first run. Since scarcely enough ammonia is present to improve the nutritional conditions of the plants, some stimulatory action may be involved. Fromm (1943) has reported such action on cultures of duckweed (*Lemna minor* L.). Possibly—to judge from the increased growth in the later runs—this chemical decomposes in the soil, and the products of decomposition (presumably nitrate and sulfate) act as nutrients to plants growing there. Sulfamate seems to lie between thiocyanate and chlorate with respect to breakdown in the soil. From its lack of response to soil type and its behavior in field plots, it appears not to be retained in the soil, but to move with soil moisture. It should therefore, like ammonium thiocyanate, find its most logical use in controlling annual and shallow-rooted perennial weeds in turf and pasture lands where the crops can avail themselves of the fertilizer elements provided. Likewise, sulfamate should be so applied that it comes immediately into contact with the plant tissues to be killed, a minimum of time being allowed for loss in toxicity by decomposition.

Reports have indicated that ammonium sulfamate spray is particularly effective against poison ivy, poison sumac, chokecherry, and other shrubs (Yeager and Calahan, 1942; Palmiter and Hildebrand, 1943; Steinbauer and Steinmetz, 1944). Descriptions of the results imply that the toxic effects may be translocated from the region of actual contact to structures below the soil surface. Since most of these reports emanate from regions of frequent summer rains, leaching into the root zone may perhaps play an important part in the effects noted. In California, tests with sulfamate and diesel oil on large, climbing poison oak (*Rhus diversiloba* T. and G.) showed no significant difference between the two herbicides when examined one year after the application, and neither gave a complete kill of roots. On small plants (under 2½ feet in height) the sulfamate killed the crowns and roots; the diesel oil killed only the stems, and the plants resprouted.

Dinitro-o-cresol.—Salts of dinitro-*o*-cresol and other substituted phenols are coming into use as selective and general contact herbicides. For this reason

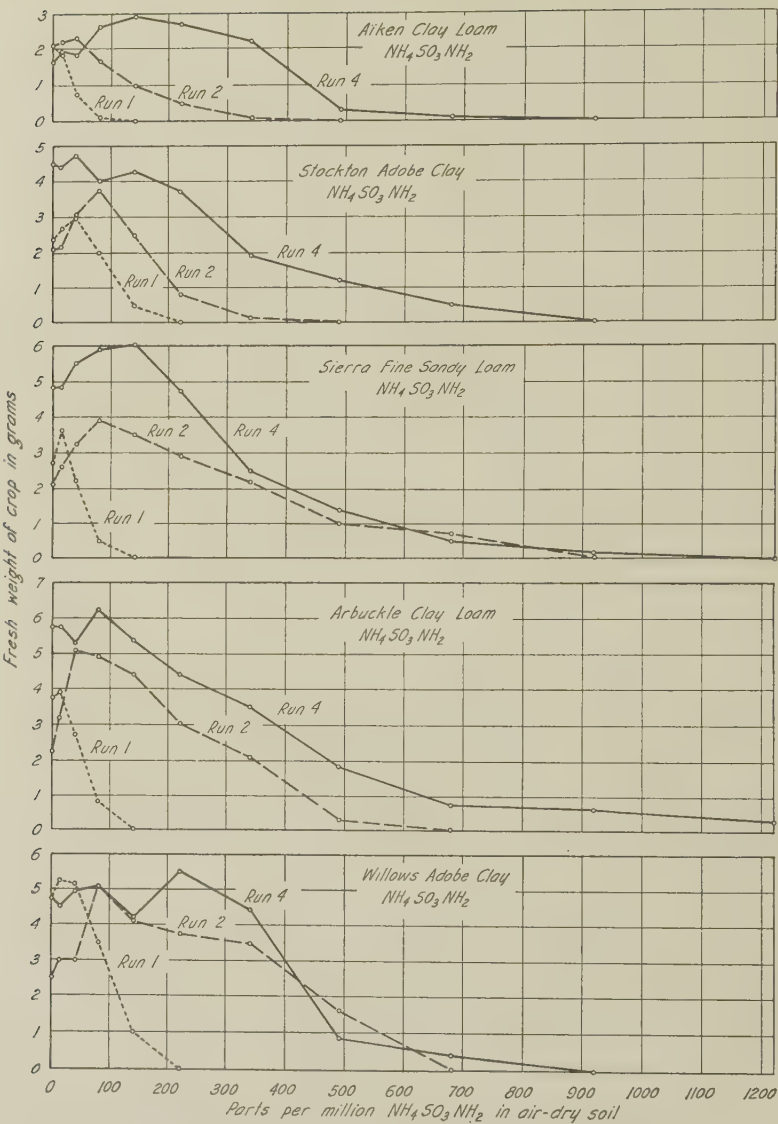


Fig. 8.—Toxicity of ammonium sulfamate to indicator plants in Aiken clay loam, Stockton adobe clay, Sierra fine sandy loam, Arbuckle clay loam, and Willows adobe clay.

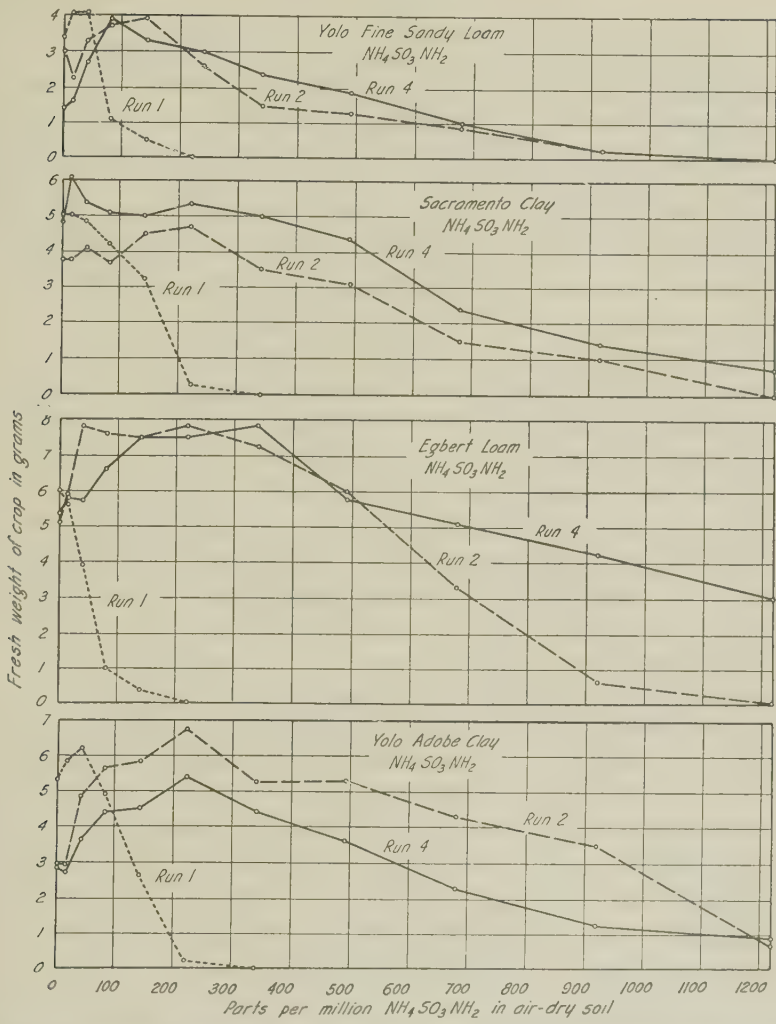


Fig. 9.—Toxicity of ammonium sulfamate to indicator plants in Yolo fine sandy loam, Sacramento clay, Egbert loam, and Yolo adobe clay.

it is necessary to know their fate in the soil, their toxicity levels, and the general relations between toxicity and soil characteristics. Studies have been made with Sinox, a proprietary herbicide containing sodium dinitro-*o*-cresylate as its active ingredient; Elgetol, a dormant spray material containing sodium dinitro-*o*-cresylate and a wetting agent; ammonium dinitro-*o*-cresylate; and dinitro-*o*-cresol in two forms.

The most extensive experiment compared the toxicities of sodium and ammonium dinitro-*o*-cresylates and dinitro-*o*-cresol used pure and used in a

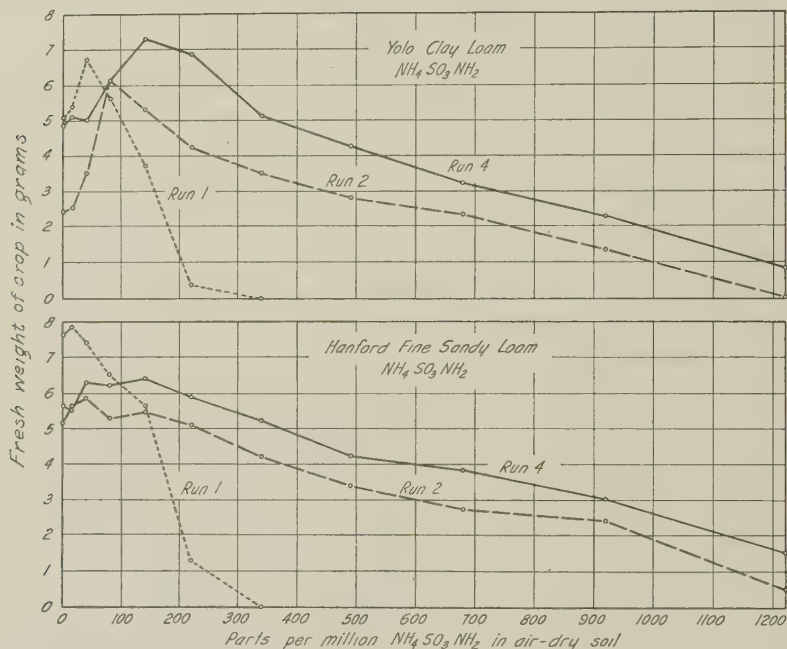


Fig. 10.—Toxicity of ammonium sulfamate to indicator plants in Yolo clay loam and Hanford fine sandy loam.

50:50 mixture with bentonite. The chemicals were added, in equimolecular amounts, to Yolo clay loam, Yolo fine sandy loam, Sierra fine sandy loam, and Stockton adobe clay. The soils were cropped three times, with drying periods between.

According to the data from this experiment, there were no differences in the toxicities of the four different chemicals added. Evidently the dinitro-*o*-cresol molecule or its ion are correspondingly toxic in soils, and the base involved is not important.

Because there were no differences, the data from the four test series were averaged, and curves were drawn showing the average toxicity of each in four soils and for three runs. Since the original test series were in duplicate, each point on each curve represents an average of eight crop weights. Figure 11 shows the curves.

Inspection of these curves reveals the following facts: dinitro-*o*-cresol has a high initial toxicity in all four of these soils, giving practical sterility at 140

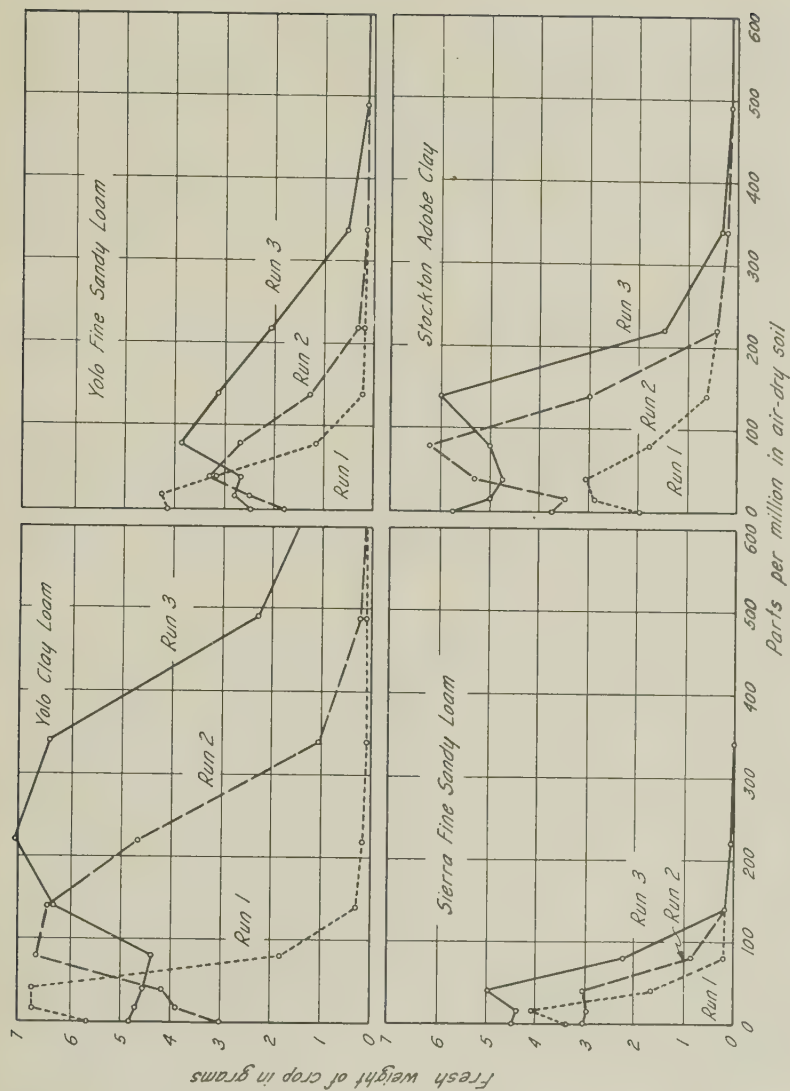


Fig. 11.—Toxicity of dinitro-o-cresol to indicator plants in Yolo clay loam, Yolo fine sandy loam, Sierra fine sandy loam, and Stockton adobe clay.

p.p.m. With certain exceptions, toxicity is highest in the light-textured soils. This fact is more readily noted in other soils than in the particular four shown in figure 11.

Toxicity decreases markedly with time and cropping, and this change is most prominent in the heavier soils. These results might suggest that dinitro-*o*-cresol is retained against leaching in soils in a form available to plants, much as arsenic is (Crafts, 1935). Percolation tests prove this to be true. Figure 12 shows the results of percolation studies on four soils.

The chemical is most firmly retained by the Yolo and Aiken soils, somewhat less by Stockton adobe clay, and least by Fresno sandy loam. Retention is less marked than with arsenic (Crafts, 1935), but more than with borax (Crafts and Raynor, 1936).

As these results demonstrate, dinitro-*o*-cresol behaves differently from chlorate, thiocyanate, and sulfamate. It is retained in the soil, depending upon the soil type, and might be expected to accumulate if sufficient leaching did not occur.

One other aspect of figure 11 is important. In the first run in all four soils, the crop at 15 p.p.m. is greater than that of the untreated check. This indicates stimulation by small amounts of the chemical. In the second runs this effect is even more prominent in three of the soils; and in the third run, after a slight initial drop, the increase becomes marked in cultures that in the first run were virtually sterile.

Of eleven soils tested with dinitro-*o*-cresol, nine show stimulation in the range of 5 to 15 p.p.m. in the first run. Of the two that fail to show it, Sacramento clay is high in organic matter; and Hanford fine sandy loam, though lighter in texture, is extremely fertile. These two soils were in a group of nine tested with Elgetol in February, 1938. They had the highest yields of any of the soils, the crops in the checks weighing 14.3 grams for the Hanford soil and 12.0 grams for the Sacramento. All soils that were run twice, except Sierra fine sandy loam, showed stimulation; and the increase in crop over the untreated checks was sometimes as much as 100 per cent.

In the curve for the third run (fig. 11) the crop yield, before rising, drops off in the region showing stimulation in the first and second runs. This may result from a tying up of nutrients in the crop in those cultures where the yield of the previous crop had been far above normal for the soil. Ultimately the yields of the third runs attained high values, indicating marked stimulation at concentrations initially toxic.

In the first analysis of the data illustrated in figure 11, curves were plotted for each chemical. Since the yields where the ammonium salt of dinitro-*o*-cresol was used did not exceed those for the sodium salt or for the parent compound, apparently the stimulation is not a straight nitrogen fertilization, at least during the first cropping. This stimulation has been noted in the field after Sinox treatment and has been attributed to partial soil sterilization. The more marked increases in yield during the second and third croppings probably involve nitrification of certain products of decomposition of the toxicant. Even here, the small amount of ammonia involved in the use of the ammonium salt had no effect upon yields.

Returning to the problem of the relation between toxicity and soil type:

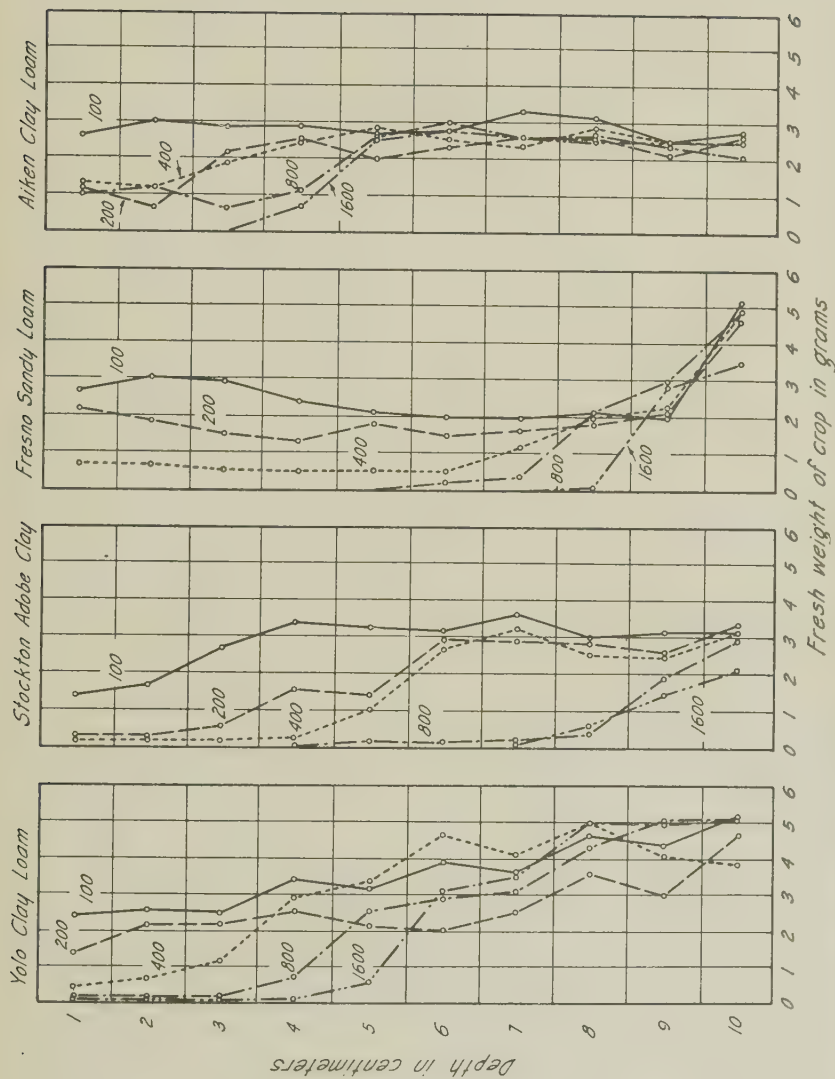


Fig. 12.—Penetration of sodium dinitro-o-cresylate into four California soils as shown by growth of indicator plants. The chemical is retained and does not move readily with soil moisture.

detailed studies of the curves for the eleven soils used show that the toxicities are affected by factors other than textural grade. If the yields in the eleven soils are expressed as percentages of check yields, so that differences inherent in the fertilities of the soil samples are compensated, the soils may be roughly grouped as follows: high-toxicity group—Sierra fine sandy loam, Fresno sandy loam, Yolo fine sandy loam; intermediate-toxicity group—Arbuckle clay loam, Stockton adobe clay, Hanford fine sandy loam, Yolo adobe clay; low-toxicity group—Yolo clay loam, Sacramento clay, Egbert loam, and Aiken clay.

Thus, generally speaking, the toxicity is highest in light soils, lowest in heavy ones. The most apparent exceptions are as follows: Hanford fine sandy loam should be in the high rather than the intermediate group; as previously mentioned (p. 474), this soil is extremely fertile, producing the highest yields of any in this study. Stockton and Yolo adobe clays should be in the low instead of the intermediate toxicity group; both are very low in fertility. The Egbert loam should have been in the intermediate instead of the high group; it is high in organic matter and very fertile.

In all the exceptions, apparently, the performance can be explained on the basis of irregular crop-yielding power. Whether this indicates (as with chlorates) a relation between absorption of dinitro-*o*-cresylate ion and nitrate ion, or whether it relates to the complex phenomenon of stimulation at low concentration, remains to be found out.

Two important aspects of dinitro-*o*-cresylate toxicity deserve consideration here. First, how hazardous is the accumulation of this chemical from successive applications as a herbicide? Second, is there any probability of its being used as a soil sterilant?

To answer the first question requires consideration of the quantitative aspects. An acre-foot of soil weighs roughly 3,600,000 pounds. To sterilize this would require for the light-textured group around 140 p.p.m. (about 500 pounds of chemical per acre foot); for the medium group 340 p.p.m. (1,200 pounds); and for the heavy soils around 680 p.p.m. (2,400 pounds). On the basis of an acre-inch the values would be roughly 40 pounds, 100 pounds, and 200 pounds, respectively.

Since 1 gallon of Sinox contains only 3 pounds of sodium dinitro-*o*-cresylate and since seldom more than 1½ gallons is applied per acre on croplands, the possibility of accumulating toxic quantities seems rather remote. Furthermore, losses of toxicity with time and cropping indicate that amounts much greater than those applied may be decomposed in the soil during each cropping. While the situation may be different in the fine sandy loam group, percolation results indicate that leaching would occur in these soils. For all practical purposes it seems very unlikely that residues of dinitro-*o*-cresol will ever become hazardous in agricultural soils. This consideration is important not only in grain crops but also in vegetable and orchard soils, where selective spraying and the use of dinitro compounds as dormant sprays are becoming more common.

A single exception to the consideration just mentioned has been noted. Where Sinox has been used as a preemergence spray (a spray applied between planting of the seed and emergence of the seedlings), light rains following the application have held the chemical in solution in a thin layer at

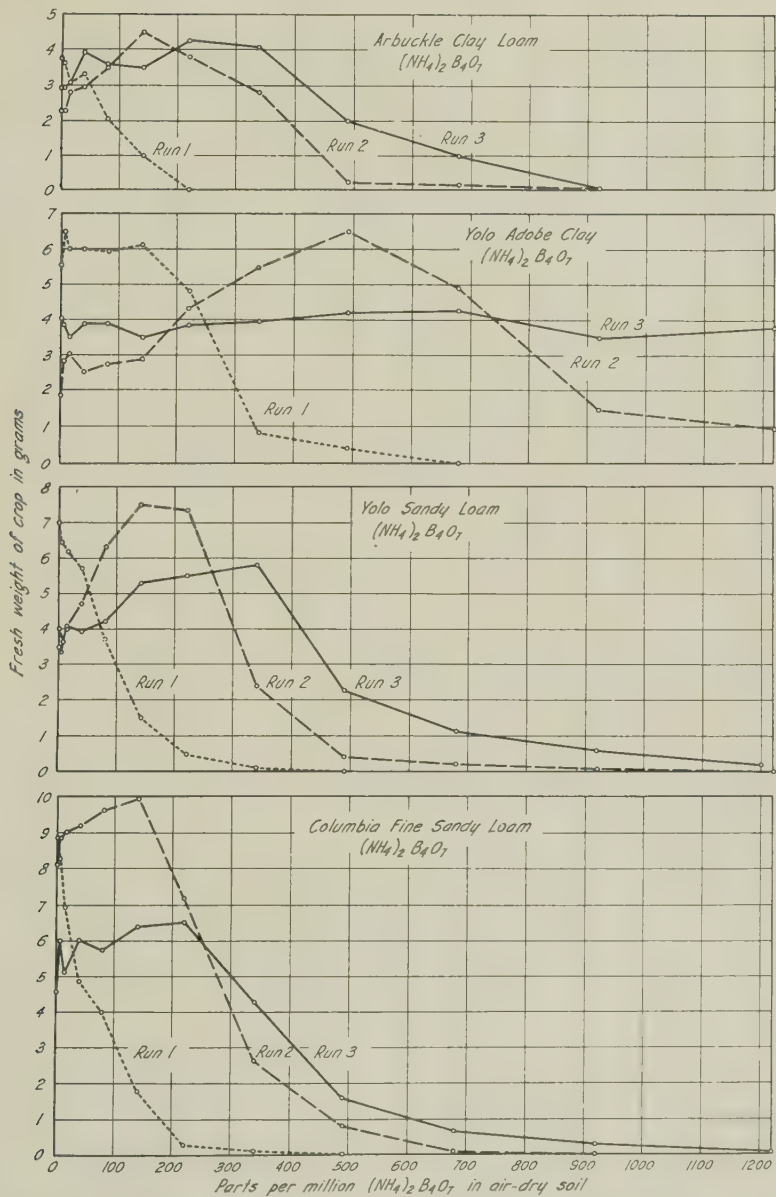


Fig. 13.—Toxicity of ammonium borate to indicator plants in Arbuckle clay loam, Yolo adobe clay, Yolo sandy loam, and Columbia fine sandy loam.

the soil surface. Onion seedlings pushing up through this moist surface soil have been seriously injured. For this reason Sinox cannot be recommended as a preemergence spray at a time when light showers are likely to occur.

As soil sterilants, substituted phenols seem unlikely to compete on a cost basis with arsenic, borax, and chlorate, three chemicals of outstanding value in this field.

Ammonium Borate.—Since the successful introduction of borax as a herbicide in the control of St. Johnswort, or Klamath weed (*Hypericum perforatum* L.) (Raynor, 1937), and as a general soil sterilant (Crafts and Raynor, 1936), the ammonium salt has been suggested as a possible substitute for use

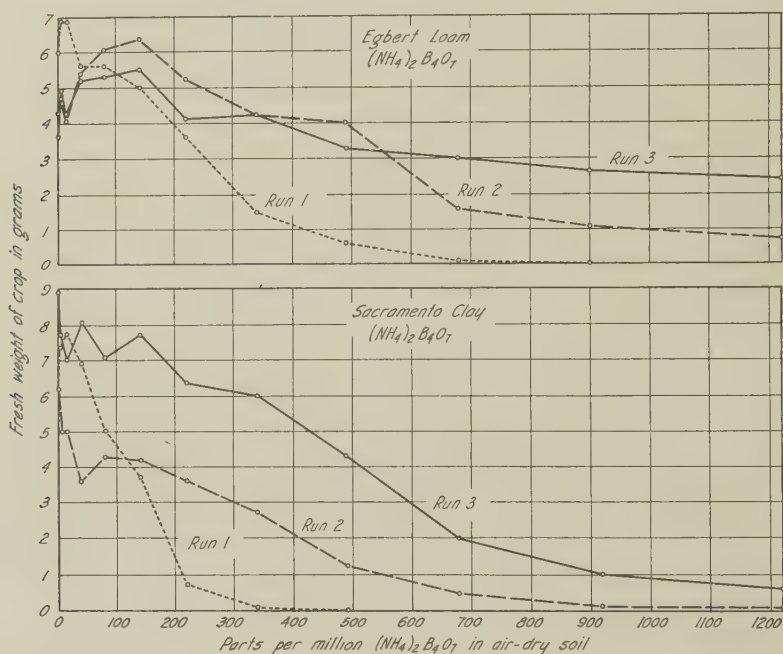


Fig. 14.—Toxicity of ammonium borate to indicator plants in Egbert loam and Sacramento clay.

on arable lands where sodium residues might injure the soil. Figures 13 and 14 show results of toxicity tests in six soils—Arbuckle clay loam, Columbia fine sandy loam, Yolo sandy loam, Sacramento clay, Egbert loam, and Yolo adobe clay. Toxicities are very similar to those for the sodium salt (Crafts and Raynor, 1936). Crop yields in general were no higher than with the sodium salt; apparently the amount of nitrogen introduced in the toxicant was not very important. The types of curves obtained indicate that the added boron affected plant growth more profoundly than did the sodium or ammonium ions.

Judging from the results of these tests, the sodium or ammonium salts of boric acid are of comparable toxicity in soils. The fertilizing effects of the ammonia involved in the ammonium salts were not significant in the soils tested. The only place where the ammonium salt might possibly prove bene-

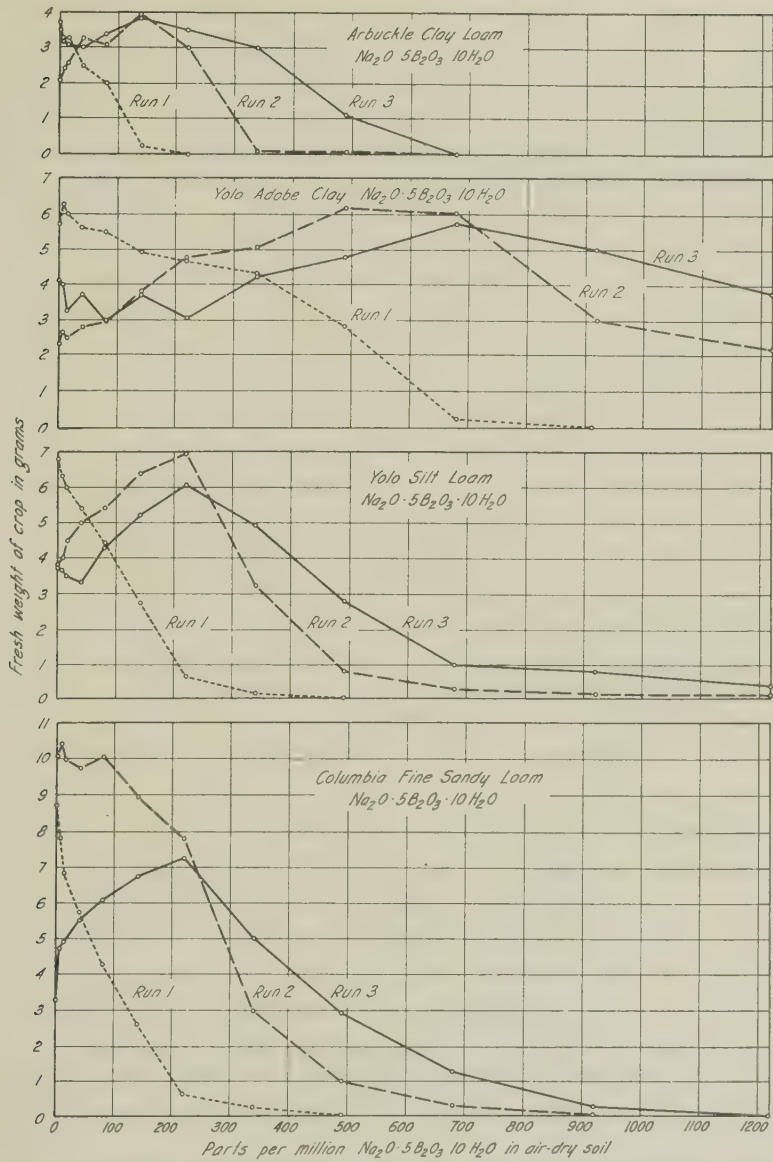


Fig. 15.—Toxicity of sodium pentaborate to indicator plants in Arbuckle clay loam, Yolo adobe clay, Yolo silt loam, and Columbia fine sandy loam.

ficial would be on arid soils already high in sodium salts. Since boron compounds are of little use as herbicides on such soils, there seems to be slight justification for the use of the ammonium salts under most conditions.

Sodium Pentaborate.—Another boron compound, sodium pentaborate, was also tested in the same six soils. The results (figs. 15 and 16) closely resemble those with borax (Crafts and Raynor, 1936). Toxicity was highest in Arbuckle clay loam; intermediate in Columbia fine sandy loam, Yolo silt loam, and

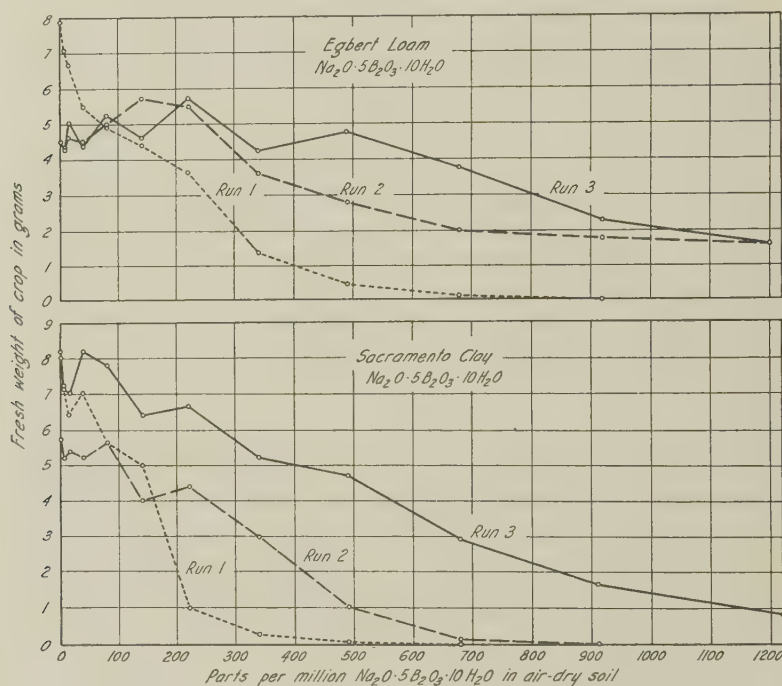


Fig. 16.—Toxicity of sodium pentaborate to indicator plants in Egbert loam and Sacramento clay.

Sacramento clay; low in Egbert loam and Yolo adobe clay. The toxicity of all boron compounds tested so far has been markedly low in adobe soils, and subtoxic concentrations have always brought about increased yield. This fact indicates a possible use of boron compounds in small amounts as fertilizers on such soils.

Toxicity of sodium pentaborate decreased with time and cropping. By the third crop, yields in soils initially sterile had become greater than those of untreated checks. Since no common nutrient element besides boron was added, and since some of the soils used were definitely not deficient in boron, these results indicate either that nutrients have been replaced and made available, or that partial sterilization has altered the relations of nutrients in the soil to increase fertility.

One other compound, ammonium persulfate, was tested. Since it proved low in toxicity, the results will not be given. It has no possible use as a soil sterilant.

DISCUSSION

As crop-production methods become more mechanized and agriculture is intensified, chemical weed control will probably assume a role of increasing importance. Methods will be aimed not only at eliminating weed species but at controlling insect and fungus pests that overwinter and increase on weeds. Preventive measures, already exemplified in the Pierce's disease control area in Tulare County and in certain citrus orchards around Redlands where sprays are being substituted for cultivation, will become widespread. When this time comes, accurate knowledge on the toxicity of all promising chemicals will be needed.

Three methods are available for general weed control: (1) permanent soil sterilization; (2) temporary soil sterilization; and (3) control by periodic spraying with translocated sprays and general contact herbicides.

To sterilize the soil permanently, arsenic is by far the most economical chemical. Methods for treating soils with it have already been described (Robbins, Crafts, and Raynor, 1942).

Of nonvolatile chemicals for temporary soil sterilization, sodium chlorate, ammonium sulfamate, and ammonium thiocyanate have been tested. Among these, chlorate persists longest in the soil, sulfamate is less persistent, and thiocyanate decomposes very rapidly. Methods for using these chemicals will relate to their toxicity and to their relative persistence, since these properties determine their effectiveness against weed species. As already mentioned, ammonium sulfamate and ammonium thiocyanate will be of most logical use against annual or shallow-rooted perennial weeds in pasture or crop areas. Their ultimate role will depend upon the success attending their application and upon the prices at which they are offered.

For use as a general contact herbicide, arsenic is hazardous because of its poisonous properties, and chlorate because it forms an inflammable mixture on weeds. Thiocyanates are corrosive to machinery. Ammonium sulfamate has been relatively expensive. Most promising have been fuel oils, solutions of substituted phenols, and emulsions of fuel oils fortified with substituted phenols.

This paper, though not concerned with the relative merits of general contact sprays, does consider the cumulative effects of the chemicals used in arable soils.

Judging from years of experience, little or no harm results from the continued use of light and medium fuel oils (distillates) as contact sprays. Weed control on highways and in citrus groves has been practiced for years with no deleterious effects upon the soils. Apparently the residues dissipate by volatilization or are consumed by soil micro-organisms.

As mentioned earlier, dinitro-*o*-cresol or its salts, used at recommended dosages, seem not to endanger the soil. During cropping they decompose in the soil at such rates that no harmful accumulation could occur in the field.

As new herbicidal materials are developed, they might well be tested by methods similar to those employed in these studies, before being given unreserved approval for general use in the field.

SUMMARY

Ammonium thiocyanate, when first applied to soils, is extremely toxic. It is not retained in an available form by the soil. Toxicity is inversely related to soil fertility and is rapidly reduced with cropping. Yields on soils sterile during the first cropping may surpass yields of untreated checks by the second or third cropping. These facts indicate that ammonium thiocyanate should be used on annual and shallow-rooted perennial weeds in cropped or pasture areas. The chemical should be so applied that it comes into immediate contact with the foliage or roots to be killed.

Sodium thiocyanate is somewhat less toxic than the ammonium salt. The toxicity of both is rapidly reduced during cropping; and yields increase, presumably because of oxidation to nitrate and sulfate. Sodium thiocyanate has not proved particularly effective in the field.

Ammonium sulfamate is lower in initial toxicity than the thiocyanate, but the change in toxicity is less marked during cropping. Toxicity is inversely related to fertility. Yields in the second and later crops showed marked stimulation above those of untreated checks. Again oxidation to nutrient ions has probably taken place.

Sodium dinitro-*o*-cresylate is toxic in soils at about the same concentration as ammonium sulfamate, and the change in toxicity with cropping is of similar magnitude. Toxicity is more definitely related to textural grade than to fertility; and this chemical is retained in an available form, particularly in soils of high colloid content. Retention is less than with arsenic but more than with borax. Initial applications in the concentration range of 5 to 40 p.p.m. greatly stimulated crop yields. This increase in yield shifted into the higher application range in the second and third croppings. Application rates, decomposition, and leaching results all indicate that no hazard exists from accumulation of dinitro-*o*-cresol or its salts in the soil after their use as selective or general contact herbicides.

Ammonium borate and sodium pentaborate resemble borax in their toxicity relations in soils. Ammonium persulfate was not toxic enough to be used in weed control.

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STUDIES ON THE ACTIVATION OF HERBICIDES

A. S. CRAFTS AND H. G. REIBER

STUDIES ON THE ACTIVATION OF HERBICIDES¹

A. S. CRAFTS² AND H. G. REIBER³

INTRODUCTION

EARLY IN 1940 Chabrolin (1940)⁴ reported that sodium pentachlorophenate at a concentration of 1.5 per cent killed wild-radish plants. Used at a dosage corresponding to 20 kg per hectare, it was effective as a selective herbicide against certain annual dicotyledonous weeds in cereals. Later in the same year Hance (1940) claimed that sodium pentachlorophenate functions as an activator with common herbicides such as sodium arsenite and sodium chlorate.

Tests in 1941 by R. N. Raynor at this station failed to indicate activation when sodium pentachlorophenate was used with sodium arsenite and sodium chlorate. The solutions were sprayed on grasses (mostly *Hordeum murinum* L.) and mixed annual weeds. Under the semiarid conditions existing, no synergism could be noted. Sodium pentachlorophenate proved toxic and, in solution, spread well on the grasses. But whenever a given concentration of sodium chlorate or sodium arsenite was applied, with and without sodium pentachlorophenate, the total toxicity of each mixture was equal only to the sum of the toxicities of the separate ingredients. Our tests varied from those of Hance (1940). We compared sodium arsenite and sodium chlorate solutions with and without sodium pentachlorophenate—that is, the concentration of arsenite or chlorate was the same in the two solutions being compared. Hance, on the contrary, compared his “activated” solutions with a so-called “standard” formula containing a higher concentration of the herbicide being tested.

CONTROLLED EXPERIMENTS ON ACTIVATION

Because many variables are difficult to control in the field, the results of the above-described field tests were checked in the greenhouse. The testing method used has since proved valuable in comparing the relative merits of many chemicals as contact herbicides. Indicator plants (both crop plants and weeds) are grown in soil in no. 10 cans. When they are large enough for testing (usually 4 to 10 inches high), each can is placed on a turntable, and the spray solution is applied with an atomizer⁵, using about 4 pounds of air pressure. The percentage injury is estimated daily until a constant condition is reached. By this method large numbers of cultures, seeded the same day and grown under the same conditions, can be treated in a short time, the type or concentration of spray solution constituting the chief variable.

In preliminary tests, sodium pentachlorophenate was about twice as toxic as sodium chlorate. In the first test on activation, a 1 per cent solution of sodium pentachlorophenate and a 2 per cent solution of sodium chlorate were

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⁴ See “Literature Cited” for complete data on citations, referred to in the text by name of author and date of publication.

⁵ The De Vilbiss atomizer no. 261 has proved suitable.

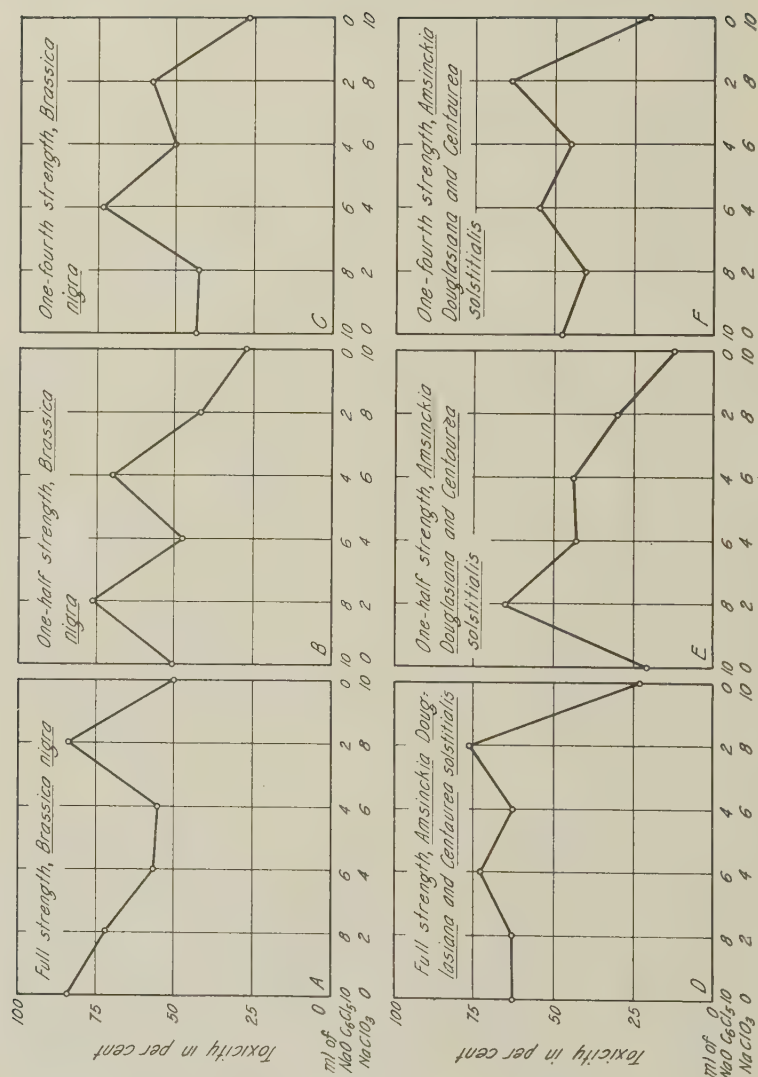


Fig. 1.—Toxicity of sodium pentachlorophenate, sodium chlorate, and their combinations to *Brassica nigra*, *Amsinckia Douglasiana*, and *Centaurea solstitialis*. Sodium pentachlorophenate concentration, 1 per cent; sodium chlorate concentration, 2 per cent at full strength. Volume ratios for the combinations below each graph.

prepared. Since 10 ml of liquid was required to wet the cultures, a series was designed to compare the toxicities of these two chemicals and their combinations. These two solutions were used in the following proportions by volume: 10:0; 8:2; 6:4; 4:6; 2:8; 0:10.

Since the total volume of solution in each case was the same and the toxicities were supposed to be approximately equal, any synergistic action should show up as an increase in toxicity above the general level determined by the two pure solutions. For purposes of comparison, the applications were made at full strength, one-half strength, and one-fourth strength of the solutions. Figure 1 shows the results. Each point on each curve represents an average of four toxicity readings made 1, 2, 4, and 6 days after spraying.

Though the graphs of figure 1 do not give undisputed evidence of synergism, one interesting feature should be noted. In every instance the toxicity values of the pure chlorate solutions are appreciably lower than those of the solutions containing 20 per cent sodium pentachlorophenate solution and 80 per cent sodium chlorate solution. Because this type of curve is characteristic of synergism, it seems possible that some activation has occurred in this experiment.

Graphs *A*, *B*, and *C* of figure 1, showing the results on black mustard (*Brassica nigra* Koch) for full, one-half, and one-fourth-strength solutions respectively, indicate that the concentration of sodium chlorate was not high enough to give a horizontal line through the points. Evidently, under the experimental conditions, sodium pentachlorophenate was three to four times as toxic as chlorate.

The fluctuations in toxicity between the six solutions of each experiment are inherent in the biological testing method. Though less uniform than desirable, comparison of results gives a measure of toxicity. Injury on fiddleneck (*Amsinckia Douglasiana* DC.) and yellow star thistle (*Centaurea solstitialis* L.) was fairly comparable with that on black mustard.

For reasons to be explained later in this paper (p. 496), the ammonium salt of pentachlorophenol is more toxic than the sodium salt. To determine whether this increased toxicity would affect the outcome of the sort of experiment just described, a second set of tests was run, using the ammonium salt. The same concentrations were used as before, and the test plants were similar. Each point on each curve is an average of 5 readings. Figure 2 presents the results.

Comparison with the graphs of figure 1 shows that, as expected, the ammonium salt of pentachlorophenol is two or more times as toxic as the sodium salt. This situation may be judged by comparing the *B* and *E* graphs (half strength ammonium salt) of figure 2 with the *A* and *D* graphs (full strength sodium salt) of figure 1. Because curves *A* and *D* of figure 2 lie within the 95 per cent toxicity level through the four highest concentrations of ammonium pentachlorophenate, it seems that the concentration of this toxicant was too high to give significant results in this type of test. Referring to figure 2, if curves *A* and *D* are examined between the fourth and sixth points, if curves *B* and *E* are examined between the third and sixth points, and if these are compared with curve *C*, the significant parts of these curves are all seen to be essentially straight lines; *F* is curved in the wrong direction to indicate activation. Hence this set of experiments yields no evidence of synergism.

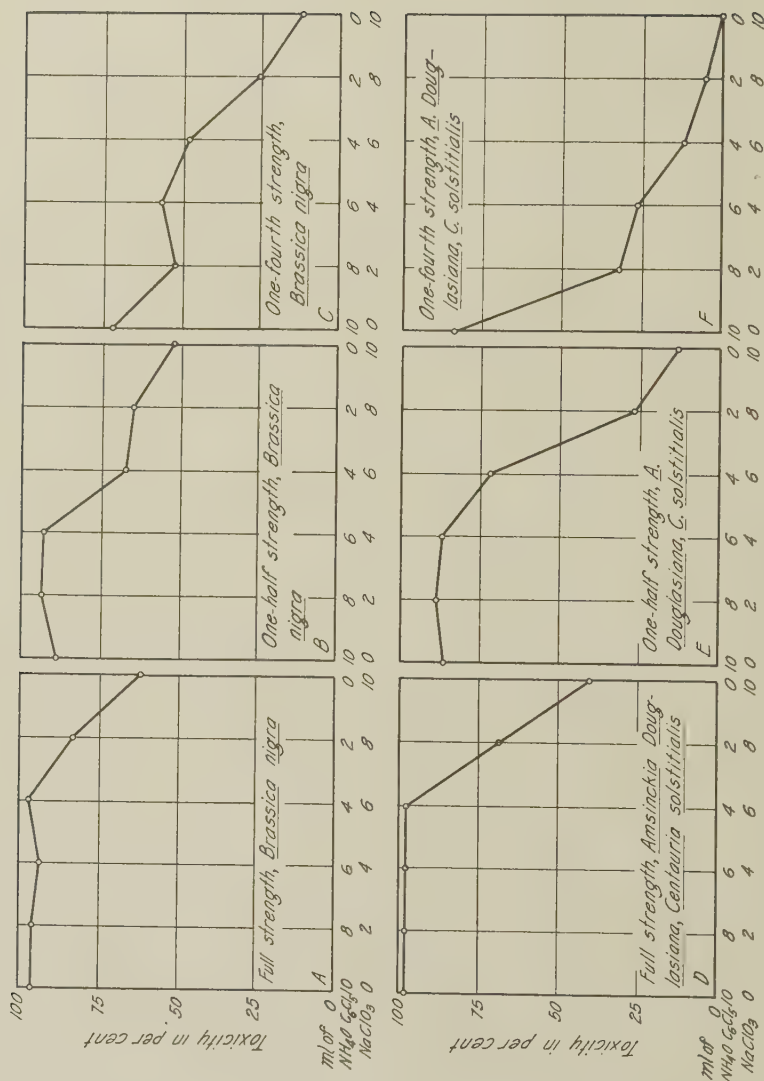


Fig. 2.—Toxicity of ammonium pentachlorophenate, sodium chlorate, and their mixtures to *Brassica nigra*, *Amsinckia Douglasiana*, and *Centaurea solstitialis*. Ammonium pentachlorophenate concentration, 1 per cent; sodium chlorate concentration, 2 per cent at full strength. Volume ratios for the mixtures below each graph.

To ascertain the effect of sodium pentachlorophenate upon arsenic toxicity, experiments were conducted with two different arsenic solutions. The first was a 0.2 per cent solution of chemically pure sodium arsenite. Since no analysis was given, the exact concentration of arsenic was not known. The second solution was 0.2 per cent As_2O_3 and was made up by mixing As_2O_3 , NaOH , and water in the proportions 4 : 1 : 3 by weight. This made a solution of sodium acid arsenite containing 50 per cent As_2O_3 by weight, which was diluted to 0.2 per cent. In each experiment sodium pentachlorophenate solution 0.2 per cent in concentration was used.

Figure 3 illustrates the results with the first solution mentioned above. The test plants were young carrot seedlings, nettle-leaf goosefoot (*Chenopodium Murale* L.) and chickweed (*Stellaria media* [L.] Cyr.). The curves are essentially straight lines. Figure 4 shows the results with the second solution, the test plants being common foxtail (*Hordeum murinum* L.). Young carrot seedlings in the pots were all injured to such an extent (95 to 100 per cent) that their values were rendered useless. According to these curves, no synergism is shown by the arsenic and pentachlorophenate mixtures. On the other hand, these experiments show that salts of pentachlorophenol are extremely toxic chemicals. The sodium salt is somewhat more toxic than sodium arsenite on the As_2O_3 basis and at least three times as toxic as sodium chlorate when used as a contact spray. The toxicity of the ammonium salt of pentachlorophenol is even greater, being approximately twice that of the sodium salt.

One further experiment was conducted using a 0.2 per cent sodium pentachlorophenate solution, a sodium arsenite solution 0.2 per cent in As_2O_3 , and a 0.4 per cent sodium chlorate solution. The three solutions were mixed in various proportions by volume and applied to cultures having young carrot seedlings and common foxtail. The carrots were completely killed in all cultures within 8 days and in all but one culture within 2 days. Results on the common foxtail plants are given in table 1.

Although the average toxicity values range from 21 to 35 per cent, there seems to be no definite pattern or trend. Again, evidence of synergism is lacking.

ACTIVATION OF SUBSTITUTED PHENOL HERBICIDES

Sodium Dinitro-o-cresylate.—Sodium dinitro-*o*-cresylate was first used as a weed spray in France in 1933. It was applied experimentally in this country during the winter of 1937-38 by Westgate and Raynor (1940) and soon gained in popularity. At present, many thousands of acres of grain, flax, and onions are sprayed with Sinox^o annually. About two years after its introduction here, experimenters found that the addition of ammonium sulfate or sodium bisulfate would greatly increase the toxicity of this organic herbicide. By using one of these relatively nontoxic salts, Harris and Hyslop (1942) increased the physiological action of sodium dinitro-*o*-cresylate in both intensity and rate. Immediately accepted by the trade, the practice of adding an activator to Sinox has become common.

Studies on the chemical mechanism of Sinox activation were started in

^o Sinox is the trade name for a proprietary selective herbicide consisting of 30 per cent sodium dinitro-*o*-cresylate and 70 per cent water.

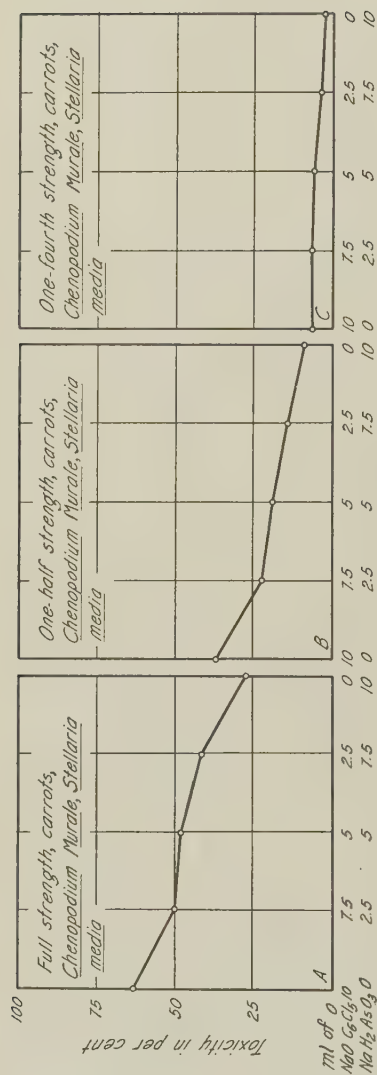


Fig. 3.—Toxicity of sodium pentachlorophenate, sodium arsenite, and their mixtures to carrots, *Chenopodium Murale*, and *Stellaria media*. Sodium pentachlorophenate concentration, 0.2 per cent; sodium arsenite concentration, 0.2 per cent at full strength. Volume ratios for the mixtures below each graph.

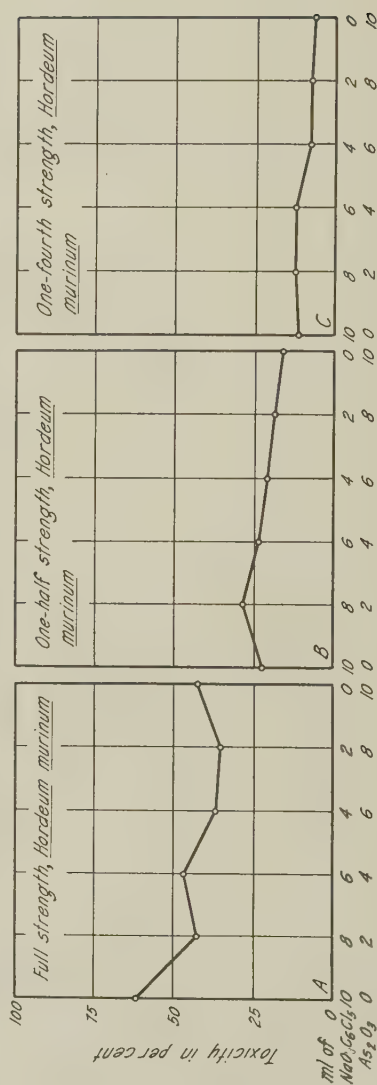


Fig. 4.—Toxicity of sodium pentachlorophenate, arsenic trioxide, and their mixtures to *Hordeum murinum*. Sodium pentachlorophenate concentration, 0.2 per cent; arsenic trioxide concentration, 0.2 per cent at full strength. Volume ratios for the mixtures below each graph.

December, 1942, and have been pursued intermittently since that time. Cultures of barley and flax with the weeds *Amsinckia Douglasiana* and *Brassica nigra* were used in the tests, and applications were made by means of the atomizer and turntable previously mentioned.

TABLE 1

TOXICITY OF SODIUM ARSENITE, SODIUM CHLORATE, AND SODIUM PENTACHLOROPHENATE ON *Hordeum murinum*, WHEN APPLIED IN VARIOUS PROPORTIONS BY VOLUME* (TOTAL VOLUME APPLIED TO EACH CULTURE 10ML)

Culture no.	Composition of solution, volume ratios			Average toxicity
	As ₂ O ₃	NaClO ₃	NaOC ₆ Cl ₅	
	per cent	per cent	per cent	per cent
1.....	10	80	10	26
2.....	10	60	30	28
3.....	10	30	60	21
4.....	10	10	80	34
5.....	30	60	10	24
6.....	30	40	30	26
7.....	30	30	40	35
8.....	30	10	60	26
9.....	40	30	30	34
10.....	60	30	10	35
11.....	60	10	30	26
12.....	80	10	10	23
13.....	33½	33½	33½	25

* Concentrations used were: sodium arsenite 0.2 per cent, sodium chlorate 0.4 per cent, and sodium pentachlorophenate 0.2 per cent.

TABLE 2

RELATION OF ACTIVATOR CONCENTRATION TO TOXICITY OF SINOX SOLUTIONS* ON *Amsinckia Douglasiana* AND *Brassica nigra*

Activator concentration in weight	Toxicity of Sinox with ammonium sulfate to		Toxicity of Sinox with sodium bisulfate to	
	<i>Amsinckia Douglasiana</i>	<i>Brassica nigra</i>	<i>Amsinckia Douglasiana</i>	<i>Brassica nigra</i>
per cent	per cent	per cent	per cent	per cent
0.000.....	14.1	12.5	14.1	12.5
0.025.....	93.3	78.0	89.0	60.0
0.050.....	93.3	85.8	94.0	84.0
0.075.....	93.3	69.1	95.0	97.0
0.100.....	93.3	92.1	97.0	97.0
0.200.....	93.3	92.6	97.0	96.4
0.400.....	93.3	90.8	97.0	97.0

* Sinox concentration was constant at 0.2 per cent. Activator concentration varied from 0.0 to 0.4 per cent.

In the first study, varying amounts of ammonium sulfate and sodium bisulfate were added to Sinox solutions, the concentration of the latter being constant throughout the series. Table 2 reports the results. The values given are the average toxicity of six successive readings made 1, 2, 4, 6, 8, and 10 days after spraying.

Each stock solution contained 4 grams of chemical to the liter of solution.

(except the highest concentration of activator, which was 30 grams per liter). All solutions were made up to a final volume of 12 ml per application.

As table 2 indicates, both salts used were very effective in increasing the toxicity of the Sinox solutions. Furthermore, in concentrations throughout the range one fourth to twice that of the toxicant, there was no significant change in toxicity. From table 2 one might conclude that the addition of one equivalent of the ammonium sulfate (66 gms) per mole of cresylate (198 gms) is

TABLE 3
TOXICITY OF ACTIVATED SINOX ON *Amsinckia Douglasiana* AND *Brassica nigra* GROWING IN FLAX

Parts by volume of 0.4 per cent Sinox to 0.4 per cent activator (final volume 8 ml)	Toxicity of Sinox with ammonium sulfate to		Toxicity of Sinox with sodium bisulfate to	
	<i>Amsinckia Douglasiana</i>	<i>Brassica nigra</i>	<i>Amsinckia Douglasiana</i>	<i>Brassica nigra</i>
	per cent	per cent	per cent	per cent
4.00 to 0.00.....	85	44	85	44
4.00 to 4.00.....	98	97	98	98
2.00 to 2.00.....	96	95	96	95
1.00 to 1.00.....	95	93	96	95
0.50 to 0.50.....	93	91	92	86
0.25 to 0.25.....	65	46	76	30

TABLE 4
TOXICITY OF AMMONIUM DINITRO-*o*-CRESYLATE ON *Amsinckia Douglasiana* AND *Brassica nigra* GROWING IN FLAX

Volume of 0.2 per cent ammonium dinitro- <i>o</i> -cresylate (final volume 8 ml)	Toxicity of ammonium dinitro- <i>o</i> -cresylate to	
	<i>Amsinckia Douglasiana</i>	<i>Brassica nigra</i>
	per cent	per cent
8.....	98	98
4.....	95	95
2.....	94	79
1.....	81	74
½.....	40	35

sufficient to bring about full activation at this concentration of Sinox. The same reasoning applies fairly well to the sodium bisulfate.

If equivalent amounts of ammonium sulfate and Sinox will give full activation, then the ammonium salt of dinitro-*o*-cresol should be as effective as the activated sodium salt. On the other hand, if excess activator above the equivalent amount provides additional toxicity, then some other factor is involved. The range of concentrations must be such that toxicities through the middle of the range are included to obtain significant differences. To test this reasoning, the experiments reported in tables 3 and 4 were performed. Ammonium dinitro-*o*-cresylate solution was prepared at a concentration of 0.2 per cent. Sinox, ammonium sulfate, and sodium bisulfate solutions were 0.4 per cent in concentration. The solutions were mixed as indicated in the tables and were made up to a final volume of 8 ml for each application.

Although no significant differences appear in the higher concentrations, at the last two rates of application, that is, in the more dilute range, the ammonium dinitro-*o*-cresylate was somewhat less toxic than the activated Sinox solutions. The activators were in excess of equivalence in all solutions. Apparently, therefore, in this concentration range something other than an equivalent amount of ammonium ion is required for full activation of the dinitro-*o*-cresylate. Furthermore, sodium bisulfate forms a white precipitate which, when applied in suspension, produces a maximum toxicity. Judging from these observations, the activation of Sinox is the effect of the free acid, or of the acid produced by the hydrolysis of the ammonium ion, and not of ammonium ion itself. Subsequently it has been proved that ammonium sulfamate, aluminum sulfate, and even sulfuric acid will increase the toxicity and rapidity of Sinox action. Sodium sulfate had no effect. Apparently any chemical

TABLE 5

TOXICITY OF SINOX AND AMENDMENTS TO CARROTS, *Brassica nigra*,
AND *Stellaria media*

Chemical and concentration	Toxicity to <i>Brassica nigra</i>	Toxicity to carrots and <i>Stellaria media</i>
	<i>per cent</i>	<i>per cent</i>
Sinox at 0.2 per cent.....	64.6	20.0
Sinox at 0.2 per cent plus NH_4OH at 0.05 per cent*....	57.1	14.2
Sinox at 0.2 per cent plus $(\text{NH}_4)_2\text{SO}_4$ at 0.2 per cent.....	89.3	83.8

* Sinox solutions containing NH_4OH above this concentration were toxic from the ammonia present.

that will provide hydrogen ions for the formation of dinitro-*o*-cresol in the spray solution will bring about activation.

Additional evidence on this problem is furnished by experiments in which ammonium sulfate and ammonium hydroxide were used in Sinox solutions. Table 5 shows the results.

As this table indicates, ammonium hydroxide repressed the toxicity of Sinox, whereas ammonium sulfate increased it. Since, furthermore, the parent dinitro-*o*-cresol dissolved in a nontoxic oil produces a solution fully as toxic, on a concentration basis, as does the activated sodium salt, undoubtedly the chemical mechanism of activation involves simply the provision of the free acid, dinitro-*o*-cresol, in the spray solution.

Table 6 presents data from a controlled laboratory experiment in which shoots of wild morning-glory (*Convolvulus arvensis* L.) were dipped in various solutions containing Sinox with ammonium sulfate, Sinox with ammonium hydroxide, plain Sinox, and ammonium dinitro-*o*-cresylate respectively. Readings of the pH values of all solutions were made with a glass electrode. After being dipped, the shoots were allowed to drain and then were set upright with their basal ends in tap water. Toxicity values as denoted by readings 1, 2, 3, 4, and 6 days after dipping are averaged in the last column.

Clearly, an alkaline reaction prohibiting the formation of the cresol in the Sinox solution reduces toxicity. The table shows a marked difference in toxicity of straight Sinox solutions and those containing the minimum amount

of activator, although the differences in pH are relatively small. This may be due to a buffering action which will be discussed later in detail (p. 498).

Sodium Pentachlorophenate.—If the fundamental behavior of all substituted phenol molecules is similar, it should be possible to activate sodium pentachlorophenate in the same way as Sinox. Table 7 presents data on toxicity tests with sodium pentachlorophenate, used alone or activated with aluminum sulfate, ammonium sulfate, and sodium bisulfate. Results are on *Chenopodium Murale* and *Centaurea solstitialis*. Toxicity readings were made 1, 3, and 8 days after treatment.

TABLE 6
RELATION BETWEEN pH VALUES AND TOXICITY OF CRESYLATE SOLUTIONS TO
Convolvulus arvensis

Chemical and concentration, per cent	(NH ₄) ₂ SO ₄	Reaction of solution	Toxicity	Chemical and concentration per cent	NH ₄ OH	Reaction of solution	Toxicity
	per cent	pH	per cent		per cent	pH	per cent
Sodium dinitro- <i>o</i> - cresylate (Sinox):				Sodium dinitro- <i>o</i> - cresylate (Sinox):			
0.20.....	0.60	6.55	98	0.20.....	0.16	11.06	57
0.10.....	0.30	6.76	94	0.10.....	0.08	10.92	24
0.05.....	0.15	7.00	70	0.05.....	0.04	10.58	13
0.20.....	0.20	6.95	93	0.20.....	0.053	10.83	61
0.10.....	0.10	7.08	86	0.10.....	0.027	10.60	24
0.05.....	0.05	7.25	73	0.05.....	0.014	10.38	11
0.20.....	0.066	7.03	94	0.20.....	0.018	10.55	55
0.10.....	0.033	7.20	86	0.10.....	0.009	10.28	28
0.05.....	0.017	7.34	62	0.05.....	0.005	10.03	11
0.20.....	7.32	56	Ammonium dinitro- <i>o</i> - cresylate:			
0.10.....	7.42	56	0.20.....	6.72	96
0.05.....	7.43	11	0.10.....	6.68	92
				0.05.....	6.70	69

As these results show, sodium pentachlorophenate can be activated by any acid salt that does not tie the toxicant up in an insoluble compound. In fact, pentachlorophenol itself is more toxic than its sodium salt, its chief drawback being its low solubility. The pentachlorophenol reported in the footnote of table 7 was a micronized product, thoroughly dispersed in water by violent shaking before application. It produced a toxicity of 66.6 per cent to *Chenopodium Murale* and *Centaurea solstitialis*. This chemical is also soluble in oil to a concentration of about 8 per cent by weight. A solution containing 0.5 per cent pentachlorophenol in any oil of proper viscosity makes an excellent general contact herbicide. An emulsion containing 4 pounds of pentachlorophenol in 6 gallons of diesel oil, made up to 100 gallons with water and stabilized with 0.80 pound of Vatsol⁷ or similar wetting agent, has proved an inexpensive general contact herbicide. A spray machine with agitator is required for application, since the emulsion is not stable enough to stand for any length of time. The ammonium salt of pentachlorophenol proved as toxic as the activated sodium salt. In concentrations ranging between 0.25 and 0.50

⁷ Vatsol is a dioctyl ester of sodium sulfosuccinate; it is a wetting and emulsifying agent.

per cent by weight, it gives promise of being a practical selective herbicide. It may be produced at a low cost, and it does not have the objectionable yellow color of dinitro compounds.

Other Substituted Phenols.—During routine testing of a great many organic chemicals, comparisons have been made between sodium and ammonium salts. In all the tests conducted so far the ammonium salts have proved more toxic than the sodium salts, and a remarkable parallelism occurs between the relative toxicities of the ammonium salts in aqueous solution and the

TABLE 7
TOXICITY OF 1 PER CENT SODIUM PENTACHLOROPHENATE SOLUTION
ALONE AND ACTIVATED TO *Chenopodium Murale* AND
*Centaurea solstitialis**

Activator concentration in weight	Toxicity with $\text{Al}_2(\text{SO}_4)_3$	Toxicity with $(\text{NH}_4)_2\text{SO}_4$	Toxicity with NaHSO_4
<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>
0.000.....	28.3	28.3	28.3
0.500.....	85.0	85.0	85.0
0.250.....	83.3	83.3	80.0
0.125.....	76.6	80.0	75.0
0.062.....	66.6	65.0	58.3

* A 1 per cent pentachlorophenol suspension in water produced a toxicity to the above weeds of 66.6 per cent.

phenols themselves dissolved in nontoxic oil. Among the phenols and the substituted phenols whose sodium and ammonium salts have been compared are dinitro phenol, dinitro-*o*-cresol, dinitro-*o*-phenyl phenol, and dinitro-*o*-cyclohexylphenol.

DISCUSSION

The activation of substituted phenol herbicides has been proved beyond question and is being employed daily in the use of Sinox and similar spray materials. Furthermore, a logical chemical mechanism to explain such activation has been postulated.

Other cases of activation are less well substantiated. Data presented in this paper offer no evidence for activation of sodium arsenite solution by sodium pentachlorophenate.

When one compares the "activated" formula of Hance⁸ (5 pounds As_2O_3 , as sodium meta arsenite, 2 pounds sodium pentachlorophenate, 2 pounds sodium chlorate, and 2 pints of wetting agent per 100 gallons) with the unactivated formula (10 pounds As_2O_3 , and 2 pints of wetting agent per 100 gallons), it seems evident, considering the toxicities of the separate ingredients, that a simple summation of the individual toxicities should approximately balance. If one were to add a pound of ammonium or aluminum sulfate to this formula, plus enough sulfuric acid to make it very slightly acid, activation of the pentachlorophenate should considerably increase its toxicity, as occurred in the experiments reported in table 6.

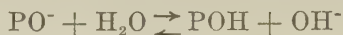
Concerning the activation of chlorate, the evidence is less clear. According to Lancaster (1942), several metallic salts that act as catalysts in oxidation processes increase the rate of killing by sodium chlorate as a contact herbicide.

⁸ F. E. Hance. Private correspondence, 1941.

Vanadium pentoxide was the best of the group. Whether sodium pentachlorophenate would act in a like capacity is not known.

When one views the problem from the reverse angle, it seems possible that the sodium chlorate might be activating the pentachlorophenate. Decision on this matter, however, must await further research.

In studying the chemical mechanism of Sinox activation, one sees that reaction of the spray solution alone is not sufficient to control the process. Considering the results given in table 6, the differences in pH between the Sinox solutions with equivalent amounts of ammonium sulfate and sodium dinitro-*o*-cresylate and those with no activator are not significant; yet the activated solutions were much more toxic. The explanation comes in a consideration of the reaction on the plant. In case of the unactivated solution, the dinitro-*o*-cresol formed by the hydrolysis of the cresylate ion may be selectively absorbed by the plant. Such absorption, removing the cresol, results in further hydrolysis, as shown by the following equation



where PO^- and POH are the substituted phenylate ion and phenol respectively. The increasing alkalinity of the solution thus limits the concentration of the phenol in the solution.

In the activated solution, containing either an acid salt (NaHSO_4) or an ammonium salt, the hydroxide ion produced combines with the acid or with the ammonium ion and thus allows further production of the cresol. Buffering may also be accomplished by addition of a strong acid or a strongly acid salt. In this case, when the pH goes below a value of about 5.2, a finely divided white precipitate of dinitro-*o*-cresol is formed; and as long as this solution remains saturated, activation is maintained.

It has been postulated that dinitro-*o*-cresol is more soluble than its salts in the plant cuticle, and therefore enters the plant more readily. The wide variety of plants responding to activated Sinox solution indicates, however, that something more fundamental is involved. Possibly the entrance of the toxicant into the protoplasm and its reaction with the protoplasm are somehow conditioned by its molecular state.

Since a Sinox solution remains activated in the region of neutrality, provided ammonium sulfate is present, it seems evident that activation is determined primarily, not by the concentration of dinitro-*o*-cresol in the solution, but by the selective absorption of its molecules by the plant. If the concentration were the only problem involved, one would expect activation to occur only in the region of pH 5.2 or below.

Two important practical applications may be derived from these chemical studies on Sinox activation. First, in order to insure maximum activation in the field, one may add an equivalent amount of ammonium sulfate (about 1 pound per gallon of Sinox) and then (using the cheapest source of acid available) acidify the solution until a slight precipitate occurs. For convenience sodium bisulfate is probably best, though sulfuric acid could be used. By this method the ammonium salt acts as a buffer, and the formation of a white precipitate serves as an indicator of the proper reaction. An alternative method would be to use acid alone, allowing the dispersed dinitro-*o*-cresol to

act as a buffer. If, however, acid in excess of the capacity of the cresol were added, the result might be a corrosive mixture that would ruin the spray rig. Another method would be to use several equivalents of ammonium sulfate. This would be somewhat more expensive.

The second point refers to the use of the ammonium salt of dinitro-*o*-cresol or any other substituted phenol herbicide. Conceivably, since such a small amount of toxicant is required, in extremely alkaline water the buffer capacity of the ammonium ion might be exceeded; the alkalinity would repress absorption. The addition of sodium bisulfate until the first appearance of the white precipitate would insure the treatment against failure.

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MOVEMENT OF CARBON DISULFIDE VAPOR IN
SOILS AS AFFECTED BY SOIL TYPE,
MOISTURE CONTENT, AND
COMPACTION

H. A. HANNESSON

MOVEMENT OF CARBON DISULFIDE VAPOR IN SOILS AS AFFECTED BY SOIL TYPE, MOISTURE CONTENT, AND COMPACTION^{1, 2}

H. A. HANNESSON³

INTRODUCTION

THE INVESTIGATION of the movement of carbon disulfide vapor in soils was undertaken by Hagan⁴. This study is a continuation of Hagan's work. He devised and reported the method, and presented sample data on Yolo soils. This paper involves soils in six series in both the dry and the moistened conditions. It deals with compaction in a more practical way than did Hagan's paper. It gives more complete data on the effects of high and low soil moisture on permeability to carbon disulfide vapor. And it compares soils of similar texture in different soil series in both the dry and the moist condition.

Carbon disulfide vapor, being extremely toxic to plant life even at relatively low concentrations, has been used extensively as a herbicide, particularly in controlling such deep-rooted perennial weeds as wild morning-glory (*Convolvulus arvensis* L.). Although in most instances it has proved effective, the results are sometimes unsatisfactory. The purpose of this study is to throw more light on the factors that influence the behavior of carbon disulfide when injected below the surface of the soil.

Being a gas at ordinary temperatures, carbon disulfide moves throughout the soil. Several factors determine its distribution: differences in partial pressure of the gas from place to place in the soil, soil texture and structure, compaction, moisture content, and soil temperature. In addition, the soil has the capacity to sorb, or take up, carbon disulfide vapor. Obviously, under field conditions these factors must be considered together, because all act interdependently to condition the over-all behavior of the vapor in the soil. Since the numerous factors involved in the open field are beyond control, the study has been conducted under laboratory conditions. Although the natural soil is disturbed by removal from the field, the problem has been somewhat simplified by using laboratory procedures.

Since liquids and solids can sorb some gases and vapors, or can form new compounds with them, two processes influence the movement of carbon disulfide vapor in the soil: sorption, tending to hold the chemical and restrict movement; and diffusion, tending to move it. Since the method used in these studies measured the net movement of vapor after sorption had come to equilibrium, diffusion alone was involved. A study of sorption as a process distinct from diffusional movement is contemplated at some future date.

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² This investigation was inaugurated and directed by the Division of Botany, College of Agriculture, under their weed-control project. The work was supported by funds contributed to the California Agricultural Experiment Station by the Wheeler, Reynolds, and Stauffer Chemical Company.

³ Research Assistant in Botany; resigned August, 1941.

⁴ Hagan, R. M. Movement of carbon disulfide vapor in soils. *Tilgardia* 14(2):83-118. 1941.

METHODS AND SOILS USED

Briefly, the method consisted in measuring the amount of carbon disulfide vapor that diffused through a soil column in a given time when one end of the column was subjected to saturated vapor and the other kept free of vapor by moving air. The total pressure of gases in and around the soil remained constant; the partial pressure of carbon disulfide vapor varied from saturated for the operating temperature to approximately zero. The vapor was concentrated at the lower end of the column; upward movement was measured after diffusion through the column reached a steady state. All experiments were conducted at controlled temperatures. Values obtained were used to calculate the permeability of the soil. For further details of the method, see Hagan's paper⁶.

Data will be discussed for several important soils occurring in the Sacramento and San Joaquin valleys of California. These soils have been derived from sandstones and shales, basic igneous, granitic materials, mixed alluvium, and organic materials.⁶

Yolo Series.—The Yolo soils are formed of transported materials derived originally from sandstones and shales, with minor mixtures from other sources. The flood plains and confluent fans occupied by these soils make up broad alluvial plains. The soils have been rapidly built up by the deposition of alluvial material in the valleys by minor streams. Weathering is relatively slight. The reaction is nearly neutral; the organic-matter content is low; and if the soil is tilled when wet, a plow sole is readily formed. The surface soil is brown, being darker than the subsoil. The entire profile is without structural development. The soils have high water-absorbing and water-holding capacities.

Hanford Series.—The soils of the Hanford series are formed from well-mixed transported material from granitic sources, carried by streams to the valleys and deposited as broad confluent fans and flood plains. The surface soils are brown to light brown, highly micaceous, generally gritty, devoid of profile development, low in organic matter, and neutral in reaction.

Fresno Series.—The material from which the Fresno soils are derived has been transported from granitic sources and deposited as nearly flat plains, rather poorly drained. Weathering under these conditions, the soils have a definite lime hardpan of irregular thickness, which occurs generally within a depth of 30 to 60 inches from the surface. The surface soils are gray to brownish gray, rather shallow, more or less calcareous, and basic in reaction; the subsoils are more alkaline and calcareous. To depths of 4 to 10 inches the surface soils are very porous and break up into granular tilth. In places the salt content is high.

Salinas Series.—The Salinas soils, formed from mixed alluvial material, occupy low terraces and stream-valley plains. The surface soils to depths of 12 to 24 inches are dark grayish brown, friable, and rather easily maintained in good tilth. The subsoils are heavier-textured and somewhat lighter-colored than the surface, with lime accumulation from place to place.

⁶ Hagan, R. M. Movement of carbon disulfide vapor in soils. *Hilgardia* 14(2):83-118. 1941.

⁶ Shaw, C. F. Some California soils and their relationships. Univ. of California Syllabus JD: 1-117. (Mimeo.) 1937.

Aiken Series.—The Aiken soils occur widely in mountain and foothill regions of the Sierra Nevada, the Cascade Range, Trinity Mountains, and Coast Ranges, occupying gentle to steep slopes. They are derived from basic igneous rocks, primarily andesites or basaltic lavas. The surface soils are brownish red to reddish brown, granular, soft in consistency to depths of 4 to 10 inches, and usually slightly acid in reaction. The subsoils are deep red and of heavier texture than the surface; they break up into soft to firm clods or lumps, coated with brownish-red colloid.

Stockton Series.—The Stockton soils are derived from mixed transported material, mainly from basic igneous rocks. They occupy basinlike flat or gently sloping plains, on which water accumulates during the rainy seasons.

TABLE 1
SOIL PERMEABILITIES AS INFLUENCED BY TEXTURE

Soil series and texture	Moisture content		Permeability units*	
	Dry	Wet	Dry	Wet
	<i>per cent</i>	<i>er cent</i>	$K \times 10^{12}$	$K \times 10^{12}$
Yolo series:				
Fine sandy loam.....	3.1	17.9	9.70	1.20
Loam.....	3.6	22.0	8.10	1.10
Clay.....	5.8	31.3	6.70	0.80
Salinas series:				
Fine sandy loam.....	3.1	16.9	8.40	1.89
Clay.....	8.9	30.5	6.95	1.51
Hanford series:				
Fine sandy loam.....	2.1	17.7	7.60	3.75
Loam.....	3.6	19.6	7.40	2.78

* The permeability unit has been defined as the gram-poisees per cm^2 per second per millimeter of mercury difference in partial pressure per cm.

An impervious substratum underlies these soils, which are already of low permeability. The surface soils to depths of 10 to 15 inches are dark gray to black, usually heavy-textured, and of an adobe structure. When wet they are plastic and sticky, but when dry they crack and shrink to large blocks with wide cracks between them. On secondary cracking they break up into granular or small-lump structure. They are high in colloidal organic material and are basic in reaction, though noncalcareous. The subsoil is heavier-textured than the surface soil, is highly calcareous in its lower part, and rests abruptly on a yellowish or brownish sandy clay substratum, more or less rocklike.

EXPERIMENTAL DATA

In this study factors that bear most directly on the use of carbon disulfide as a herbicide have been emphasized.

Texture.—In table 1 are presented data for several textures: air-dry⁷ samples were compacted to an apparent density of about 1.20 grams per ml. The highest flow of carbon disulfide is obtained in the fine sandy loams, the lowest in the clays; and an intermediate flow in loams. When these same soils

⁷ The terms "dry" and "wet" as used in this paper refer to approximately an air dry condition, and to a water content near the moisture equivalent. Tables 1, 2, and 3 give the moisture contents of soils reported.

are moistened to nearly their moisture equivalents the permeabilities, though very low, are again highest in the fine sandy loams and lowest in the clays.

A similar textural relation was found to hold at both higher and lower compaction values as well. One must bear in mind, when appraising the effect of texture on the permeability of soils to carbon disulfide, that it becomes difficult to separate the effect of the inherent structural characteristics.

Compaction.—In tillage operations, soils may be compacted by the application of forces normal to the soil surface (compression), or by the combination

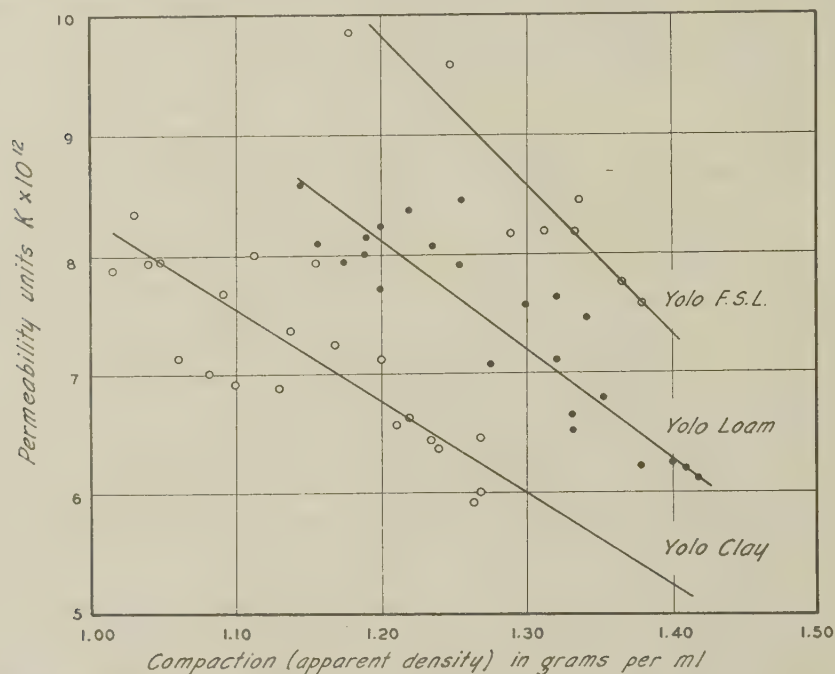


Fig. 1.—Effect of compaction (apparent density) on permeability of air-dry soils to carbon disulfide. Curves were drawn in by approximation; moisture contents of these samples were comparable with those reported in table 1.

of forces acting normal and parallel to the surface (shearing). Strictly speaking, both forces are acting simultaneously in all types of field compaction; but their relative magnitudes may vary greatly, thereby altering tremendously the permeabilities of field soils. Samples were prepared first in which the permeabilities of the soils were altered by varying the compression force applied. These are more or less simulated field conditions in which compaction has been produced by the application primarily of weight (such as heavy machines) on the top of the soil. In figure 1 the effect of compaction of the air-dry soil in this manner is shown by the curves that were approximated for three different textures. In all cases as the compaction (apparent density) was increased, the permeability was materially reduced.

The effects of compaction produced by compression and by shearing forces were tested on Dinuba sandy loam. One sample was compacted air-dry by a

compressional method and its permeability measured. It was then moistened to near its moisture equivalent and its permeability determined again. A second sample was compacted while in the moist (approximate moisture equivalent) condition by working it with a spatula. Its permeability was determined. It was then dried to the air-dry condition and its permeability again measured. The results were as follows:

Dominant compaction force	Permeability when air-dry (about 2 per cent), $K \times 10^{12}$	Permeability when moist (about 12 per cent), $K \times 10^{12}$
Compression	7.50	1.70
Shearing	0.95	0.15

Both samples compacted by shearing had low permeability; in fact, the puddled sample was almost impermeable to the vapor.

Moisture.—Permeability measurements on soils were limited to samples in the air-dry condition and moistened to a water content of approximately the

TABLE 2
PERMEABILITY OF THREE TEXTURES OF YOLO SOIL TO CARBON DISULFIDE VAPOR
AT TWO MOISTURE CONTENTS

Soil series and percentage moisture content	Permeability unit $K \times 10^{12}$	Soil series and percentage moisture content	Permeability unit $K \times 10^{12}$
Yolo fine sandy loam:		Yolo loam (<i>Continued</i>):	
Low moisture content:		High moisture content:	
3.10.....	9.63	19.10.....	3.06
3.16.....	9.78	21.10.....	2.50
High moisture content:		22.60.....	0.64
15.60.....	2.25	22.40.....	0.67
17.70.....	0.66	23.50.....	0.33
18.10.....	1.50	Yolo clay:	
18.80.....	0.69	Low moisture content:	
19.90.....	0.23	5.75.....	7.12
Yolo loam:		6.10.....	6.24
Low moisture content:		6.10.....	6.37
3.05.....	7.43	High moisture content:	
3.75.....	8.00	31.30.....	0.67
3.75.....	8.20	31.40.....	0.90
4.20.....	7.73	32.30.....	0.31

moisture equivalent. Intermediate moisture contents of uniform distribution within the columns could not be obtained. Yolo soils of three different textures were used; data reported have been selected from many determinations on a compaction basis, only those of approximately 1.20 grams per ml being given. Results are presented in table 2.

At high moisture contents the permeabilities of all types are greatly reduced; in fact, some are almost impermeable. Yet about one fourth of the volume of each soil column is still occupied by gases. The nonsolid volume of the soil is apparently occupied by gases and water in approximately equal amounts.

Soil Series.—Several series of similar textures were compared. For a more complete picture of their permeabilities to carbon disulfide, soils at both low and high moisture contents are reported in table 3.

Permeability data are presented for Yolo, Salinas, and Hanford fine sandy

loams, air-dry and moistened. The Hanford soil under the experimental conditions had a lower permeability than the Salinas and Yolo soils when dry; when wet Hanford has the highest value.

The Egbert, Yolo, Fresno, and Hanford loams are compared. Again the Hanford soil had the lowest permeability when dry, but the highest when moist, whereas the other series have values of about the same magnitude. When wet the Hanford is substantially more permeable than Egbert, Yolo, or Fresno soils of this texture.

Values for three clays are also given, indicating a lower permeability for the Stockton series than for the Salinas or Yolo clays.

TABLE 3
PERMEABILITY OF SEVERAL CALIFORNIA SOILS* TO CARBON DISULFIDE VAPOR
AT TWO MOISTURE CONTENTS

Soil texture and series	Moisture content		Permeability units	
	Dry	Wet	Dry	Wet
	<i>per cent</i>	<i>per cent</i>	$K \times 10^{12}$	$K \times 10^{12}$
Fine sandy loam:				
Yolo.....	3.1	18.8	8.22	0.70
Salinas.....	3.1	16.9	8.42	1.89
Hanford.....	1.7	17.0	7.58	3.75
Loams:				
Egbert.....	7.7	32.2	7.90	2.25
Yolo.....	3.4	22.6	7.94	0.64
Fresno.....	3.2	15.9	8.43	2.18
Hanford.....	3.6	18.5	7.30	2.78
Clay:				
Salinas.....	8.9	30.5	6.95	1.51
Yolo.....	6.1	31.3	7.11	0.67
Stockton.....	7.1	27.1	5.60	0.60

* All soils were compacted at an apparent density of 1.20 grams per ml.

Temperature.—The effect of soil temperature on the movement of carbon disulfide vapor through the soil is discussed by Hagan⁸. In brief, as the temperature changes, the result is to change the partial pressure of the vapor, with the net result that for each 10-degree-C rise in temperature the movement of the vapor is increased by about 50 per cent.

DISCUSSION

Samples of surface soils of several types were obtained from widely separated locations in the Sacramento and San Joaquin valleys. Admittedly, preparing these soils for laboratory investigation has altered their natural condition by destroying their structural development to such an extent that the larger structural units no longer exist. This investigation involves both surface and subsoil, and the problem is concerned primarily with the soils under cultivation. The present method offers a satisfactory treatment of the surface soil. To gain further information on the permeability of subsoils, another technique has been developed, which will be discussed in a later paper.⁹

⁸ Hagan, R. M. Movement of carbon disulfide vapor in soils. *Hilgardia* 14(2):97–102. 1941.

⁹ Hannesson, H. A., R. N. Raynor, and A. S. Crafts. Herbicidal use of carbon disulfide. (In press.)

According to the results of this investigation, the most important factors that may influence the movement of carbon disulfide are, first, the moisture content of the soil; second, the kind of compaction that produces a puddled condition; third, the temperature of the soil. One must consider all three factors collectively in understanding the movement of this gas within the soil under field conditions. To a lesser degree, soil texture may effect the movement. As the clay content increases, the permeability of the soil decreases in the samples prepared and studied under laboratory conditions. Since, however, the natural structure of the soil has been disturbed by the treatment, some allowance must be made when applying this conclusion to subsoil conditions unaltered by cultivation or to a soil profile in its natural state. A further conclusion is that no great difference in permeability of surface soil to carbon disulfide exists between the soil series studied except for minor differences, probably associated with moisture-holding capacities. One exception may be cited: of particular interest was the finding that permeability of the Hanford soils was highest of all the soils tested in the moist condition.

In the West, the formation of plow sole or plow pan after cultural practices is a serious problem. The data presented show that the permeability of the Dinuba sandy loam when puddled and wet approaches a zero value. They further indicate that even when the soil has been reduced to the air-dry condition the permeability value is very low.

The plow sole or plow pan occupies a critical position in the soil profile. Its ability to restrict the downward movement of carbon disulfide vapor toward the deep-lying roots is very significant.

Perhaps no single factor more affects the permeability of a laboratory-prepared soil to carbon disulfide than does moisture: as water is added to the soil, the effective conducting channels become smaller and smaller until, at a moisture content of approximately the moisture equivalent, the permeability value of the wet soil approaches zero. This fact would indicate that shortly after a soil has received an application of water by heavy rains or by irrigation, conditions for movement of carbon disulfide vapor in the tilled layer are extremely unfavorable. This suggests that injection of carbon disulfide should place the chemical beneath the tilled surface layer and that moisture content, even in the subsoil, should be low if free diffusion is to take place.

SUMMARY

Certain factors affecting the movement of carbon disulfide vapor in soils have been studied according to the method devised and reported by Hagan.

The permeability to carbon disulfide of several soil series and types from widely separated sections of the Sacramento and San Joaquin valleys in California was investigated by this method.

Under most conditions moisture is an important factor in influencing the movement of carbon disulfide vapor in soils. As the moisture content is increased, the permeability of the soil is decreased; a value of nearly zero is reached as the moisture content of the soil approaches its moisture equivalent.

Compaction, as in plow sole or plow pan, is very important in controlling the movement of carbon disulfide vapor under field conditions. If moist, such a layer may be almost impermeable to this vapor.

In cultivated surface soils, differences between textures are of less significance. The permeabilities of the lower-clay-content soils tend to be higher whether dry or wet. The effect of structure is not dealt with in this paper.

The full significance of differences in the permeability of individual soil series cannot be presented here, since only surface samples were studied in the granulated state. The results, however, indicate no great difference between soil series when in this condition; and information is being accumulated by other means on the permeability of the subsoils of several soil series in their natural condition.

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